Fabrication of C6-Fluorocarbon-Dendrimer Based Superhydrophobic Cotton Fabrics for Multifunctional Aspects

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Research Article

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Posted Date: May 21st, 2021

DOI: https://doi.org/10.21203/rs.3.rs-490421/v1

Fabrication of C6-Fluorocarbon-dendrimer based superhydrophobic cotton fabrics for multifunctional aspects

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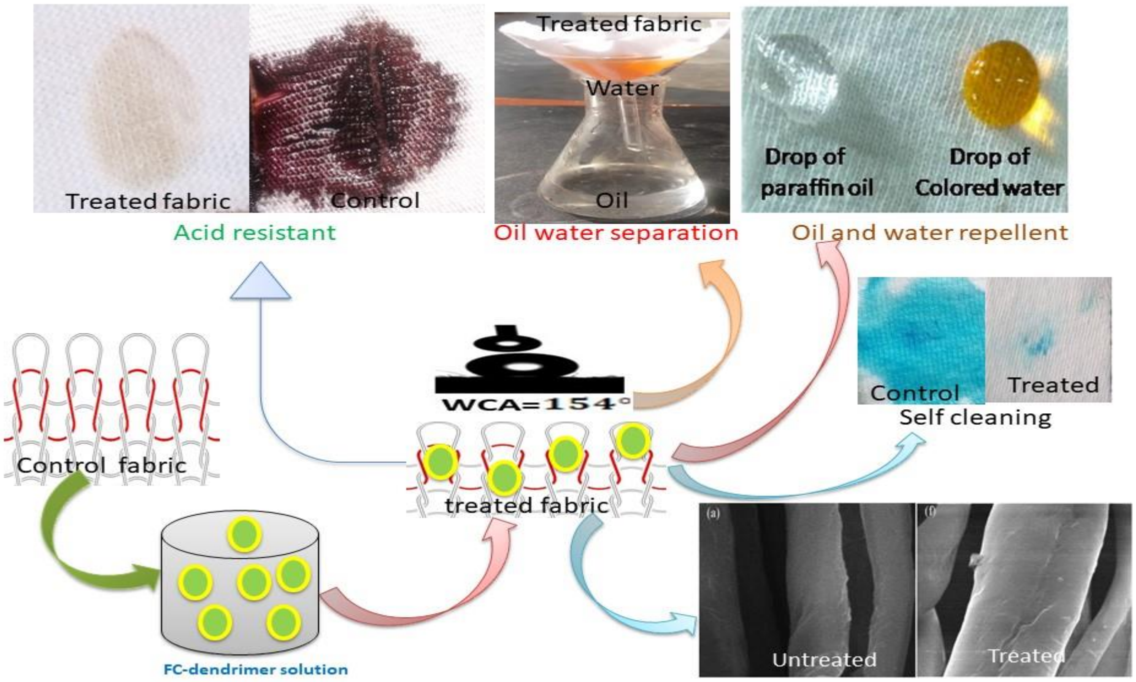
Abstract

C_6-Fluorocarbon (FC)-dendrimer has been applied on cotton knit fabric for developing water repellent, self-cleaning, oil-water separation, acid-resistant, antibacterial, UV resistant and flame retardant property while maintaining acceptable levels of comfort for wearers. The C_6-Fluorocarbon (FC)-dendrimer coated 100% cotton single jersey knitted fabric samples were prepared using "pad-dry-cure" method, and characterized and tested for comfort and other textile properties. The 90 g/L and 100 g/L FC-dendrimer treated cotton fabrics provided excellent water repellency, oil-water separation and self-cleaning performance. But air permeability and thermal conductivity were 13%, 15%, and 40%, 54% lower, respectively, than those of untreated fabrics. The presence of FC-dendrimer in the treated fabric was confirmed by FTIR, SEM, EDS and XRD analyses. SEM analysis was employed to study the morphology of deposited FC-dendrimer particles on the fabric surface. Thermal behaviors were evaluated by TGA and DTA. The FC-dendrimer-treated fabric also showed promise as a UV ray absorber, antimicrobial activity, acid resistance and flame retardant property. Overall, the result suggests that FC-dendrimer can be a valuable ingredient in the manufacture of multifunctional products.

Keywords: FC-dendrimer, cotton fabric, self-cleaning, superhydrophobic, protective textiles.

Graphical abstract



Introduction

The rapid growth in the use of protective textiles and their uses has created a serious need for the application of functional finishes. Recent market surveys have shown that apparel consumers all over the world are demanding functionality in their purchased products. Some of the best examples of protective and functional textiles are those which are water-resistant, oil repellent, soil repellent, acid-resistant, flame-retardant, fade-resistant, UV resistant, microbial resistant, wrinkle-resistant, and so on. "Protective" textiles are garments or fabrics which protect the wearer from environmental hazards **(Gupta 2000).** Two qualities that outdoor garments require are water repellence and oil repellence. These, plus the ability to prevent dry dirt and soil from attaching to the fabric, are also needed for home and factory wear. Yet the clothing should always remain comfortable. Army and police wearers need protective clothing to be lightweight and not to inhibit their freedom of motion **(Jiang et al. 2017).**

A Variety of chemicals are used to develop different functional properties. Such as, a dispersion of benzotrizole-type derivative and fluorocarbon resin-type compound are taken for UV-resist and water-repellent finishes **(De et al. 2005).** A fluoropolymer was used to impart the liquid repellent properties and 'Zydex Zycrobial' non-leaching type antibacterial agent was used to develop antimicrobial properties of woven fabric (**Midha et al. 2014).** Fluoropolymer (water repellent agent) and 2-hydroxy-4-dodecyloxybenzophenone (UV absorber) were used to reduce the fading propensity of upholstery fabric (**Crews and Clark 1990).** Chitosan and fluoropolymers were used to impart the antimicrobial and blood repellent property for nonwoven materials **(Lee et al. 1999).** The cotton fabrics treated with 1.5% $Al(NO_3)_3$ and 20 mol/L sodium stearate showed excellent hydrophobic properties and UV protection efficiency **(Pana et al. 2012).** Plasma treatment and fluoroalkyl water-born siloxane (FAS) were used to impart super-hydrophobic, oleophobic and self-cleaning properties on cotton fabric **(Vasiljevic et al. 2013).** Fluoroalkyl-functional siloxane (FAS) and 3-(trimethoxysilyl)-propyl dimethyl octadecyl ammonium chloride (SiQAC) were used to develop multifunctional properties like water-oil repellent and antimicrobial properties **(Simoncic et al. 2012).** But in this research, it has been attempted to develop different functional properties by using only one main chemical, it's is a noble approach.

Protective clothing can be achieved with many types of finishes, including paraffin, silicone, stearic acid-melamine-based compound, fluoropolymer and so on. Paraffin was one of the earliest water repellent agents. These repellent products are generally emulsions containing aluminum or zirconium salts of fatty acids, usually stearic acid. Despite providing good water repellency effects, paraffin repellents do not repel oil and are generally not durable to laundering and dry cleaning. Additionally, fabrics treated with paraffin-based finishes are less permeable by air and vapor, resulting in poor wear comfort **(Schindler and Hauser 2004).**

Silicon repellents offer a high degree of water repellency at relatively low concentrations. Yet, their repellency can be reduced if excess amounts are applied. They have only moderate durability to laundering and dry cleaning, and no oil and soil repellency. Wastewater, particularly from residual baths of the finish application processes, is toxic to fish **(Schindler and Hauser 2004).** Silicon compounds like tetraethoxysilane (TEOS) modified cotton fabric showed good water repellent properties (**Wang et al. 2007).** TEOS exhibited toxicity and may cause potent respiratory hazards **(Omae et al. 1998).**

For durable liquid-repellent finishes, Stearic acid-melamine-based repellent compounds are extensively used. Stearic acid-melamine repellent chemistries are composed of compounds formed by a reaction between stearic acid and formaldehyde and melamine. But these finishes have decreased abrasion resistance and fabric tear strength, cause changes in the shade of dyed fabrics and release formaldehyde. The release of formaldehyde is a problem for human health as well as the environment **(Schindler and Hauser 2004).**

Fluoropolymer is mostly effective for liquid repellent and soil release finishes. But the conventional C8-based fluorocarbons are suspected to release bio-persistent and toxic components perflurooctanoic acid (PFOA) and perflurooctane sulfonate (PFOS). They affect human reproduction, hormone system and cell processes. After immense research and development chemists succeeded to replace C8 chemistry with C6 by using short 6 carbon-fluoro chains. They excluded the possibility of fluorocarbon products broke down into PFOA. The compound Perfluorohexanoic acid (PFHA) obtained from C6 technology has 40 times fewer accumulative effects than PFOA (**Danish Environmental Protection Agency 2005).**

The ability of a cloth to repel water and oil depends on its roughness and surface free energy. A liquid will spread across the cloth if its surface energy is less than that of the cloth **(De et al. 2005; Coulson 2007).** The surface tension of water is 72 mN/m. So the surface-free energy of the cloth must be less than that surface tension of water. Oil has a surface tension of about 20-35 mN/m. So, to be oil repellant, the cloth must be less than that. The surface energy of fluorocarbon polymer is 10-20 mN/m. Repellent property of fluorocarbon-based chemicals has much better than silicone repellants as the surface energy of silicon repellent is 24-30 mN/**m**

(Coulson 2007; Duschek 2001; Khoddami et al. 2012). The fluoropolymer forms a covering of the fibers which reduces the surface-free energy of the textile. Such a covering also increases liquids' contact angle on the fabric's surface **(Schindler and Hauser 2004)**. On the other hand dendrimers are used to enhance water repellence by changing the nanostructure of the cloth's surface. They are a core molecule with a large number of branched organic atoms **(Windisch et al. 2000).** Recently dendrimers have also been used as antimicrobial **agents (Ghosh et al. 2010).** Fluorocarbon dendrimers are not only water-repellent but also oil- and soil-repellent, when the surface energy of the cloth has been reduced **(Sayed and Dabhi 2014). Fig. 1** shows the chemical structure of a fluorocarbon polymer on fibre surface (**Schindler and Hauser 2004).**

Fluorocarbon finished product showed good resistance to water absorption, excellent adhesion properties and better transmission of water Vapor (**Shekar et al. 2001).** Transmission of Water Vapor through fabric is the key component of thermal comfort. Thermal comfort is produced by the fabric's air permeability, water – especially water Vapor- permeability, heat permeability **(Behera and Hari 2010).** Wearing impermeable clothing can cause a 3^0 C rise in temperature within ten minutes when a person is performing a physically moderate task and a 10^0C rise in temperature when performing physically demanding work (**Day and Sturgeon 1986**). Generally, water-proof fabrics have lamination, high-density tight weaving and solid coating, all of which block air and water vapor movement. As a result, the wearer feels discomfort, due to the condensation of sweat inside the clothing. On the other hand, FC-dendrimer repellent finishes overcome these problems by developing a permeable layer that allows water vapor, heat and air to pass through the clothing. As Dendrimer macromolecule's ability to build up crystal structures in nano range on the fabric surface with the benefit of not impairing the permeability the textile to air and vapor. Then the wearer feels at ease. Such water-repellent fabrics can be used for various types of protective garments against rain, snow and cold, active sportswear, etc **(Krishnan 1991; Roey 1992; Chinta and Satish 2014; Colleoni et al., 2011).**

Namligoz et al (**2009**) found that polymeric dendrimer containing FC had better results for water, oil and stain repellency than conventional FC ones in terms of performance and washing resistance. It is assuming that FC-dendrimer provides more functional groups on the surface of the textiles than single one. On the other hand, the dendrimer is well known regular structure, built up in several generations starting from a core and containing a surface with a high density of end groups (functional groups). The number of end groups increases exponentially with the number of generations building up the dendrimer.

Fluorocarbon polymers are applied together with dendrimers, causing self-organisation where the fluorocarbon chains are enriched on the surface and co-crystallize with the dendrimers. Dendrimers are highly branched oligomers with non-polar chains forming a starbrush structure. They force the polar parts of the FC polymers to form the surface structure mentioned at the beginning of this section. The resulting polar and non-polar sandwich arrangements are highly ordered, causing equal or better repellency effects with lower amounts of fluorocarbon compared to dendrimer-free FC finishes. Other advantages include low condensation temperature (80–130 °C), high abrasion resistance, good wash permanence and soft hand (**Duschek, 2001**). A hypothetical structure of a dendrimer is shown in **Fig. 2 (Schindler and Hauser 2004).** In addition**,** there is little evidence that the hydrocarbon dendrimer is more environmentally friendly than FC polymer. Hydrocarbon dendrimer-treated fabric could withstand more water hydrostatic pressure than FC-treated fabric. The breathability of hydrocarbon dendrimer treated fabric was more than FC polymer treated fabric.

A fundamental principle regarding light interactions with matter is the fact that chemical reactions can be initiated by the energy provided by electromagnetic radiation. These types of reactions are also called photochemical reactions, which implies that light is absorbed in a chemical substance (**Appleyard, 2012**). An outdoor jacket is usually being exposed to sunlight and therefore this was an important test in order to evaluate the performance of the treated fabrics against UV radiation. After 8 cycles UV radiation, spray rating of fluorocarbon treated fabric did not deteriorate. After 16 cycles the fluorocarbon still show an acceptable spray rate of ISO 4. Fluorocarbon can handle UV exposure better than the hydrocarbon dendrimer (**Åkerblom & Göranzon, 2014**). These findings revealed that FC and dendrimer-treated fabric exhibited UV radiation protection property. But a clear mechanism did not found. It is assuming that coating chemicals absorbed UV radiation.

Under suitable conditions micro-organisms that inhabit soil, water, and air can develop and proliferate on textile materials. These organisms include species of bacteria, fungi and algae which causes a range of undesirable effects, not only on the textile itself but also on the user. Textiles made from natural fibers are generally more susceptible to bio-deterioration than are synthetic man-made fiber. However, microbial activity can be minimized by keeping susceptible materials dry, as surface growth will only occur when the relative humidity is high. So, it is expected that the hydrophobic property will be helpful for the inhibition of microbial growth.

Flame retardants are chemicals that are applied to materials to prevent the start or slow the growth of the fire**.** At present researchers do incorporate several flame retardant elements like phosphorus (P), nitrogen (N), silicon (Si) and boron (B) in the different chemical compound. In this analysis, the flame retardant property is put to the test as the dendrimer contains nitrogen (N).

Literature survey revealed that a significant number of **researches have** been already done on fluorocarbon polymer and **FC-dendrimer** for textiles as water and oil repellent chemicals, their application procedure, improved water repellency and their durability and various wash fastness **(De et al, 2005; Midha et al. 2014; Crews and Clark 1990; Lee et al.1999; Khoddami et al. 2012; Sayed and Dabhi 2014; Shekar et al. 2001; Behera and Hari 2010; Wang et al. 2011; Zhou et al. 2013; Shekar et al. 1999; Cerne and Simoncic 2004; Lidija and Simoncic 2008; Chowdhury 2018).** But almost insufficient works were found on the self-cleaning property, flame retardant property, oil-water separation, acid resistance, antimicrobial activity, UV protection factor and thermal comfort attribute of the fluorocarbon-dendrimer treated fabric.

Traditionally water-repellent chemicals have been applied to woven fabric. But woven fabric exhibits great limitations in terms of comfortability like low moisture absorbency, low air permeability and less elasticity compared to the knitted fabric. So, cellulosic knits are can be chosen for water-repellence treatment, as knitted fabric possesses a flexible, elastic structure, freedom from static, a porous surface, softness, good breathability and high absorbency, all of which maximize comfort for the wearer. Thus, in this study, fluorinated polymers are applied to cellulosic knit fabrics (**Ajgaonkar1998).**

In view of all this information, the aim of the present work is to study the different functional properties such as self-cleaning, oil-water separation, acid-resistant, antimicrobial activity, UV protection factor, flame retardant, and thermal comfort properties along with the water-oil repellent property of 100% cotton knitted fabric treated with C_6-fluorocarbon resin with dendrimers. The water-resistance properties were determined in terms of measurements of the contact angle, spray rating test, hydrostatic pressure, and vapor permeability, whereas the morphology and surface composition of treated fabrics were examined with scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis. In this work which concentration of fluorocarbon dendrimers was suitable for particular end usage also studied? Statistical analysis also involved finding out the acceptable thermal comfort properties for different concentrated fluorocarbon dendrimers treated fabric at 5% level of significance.

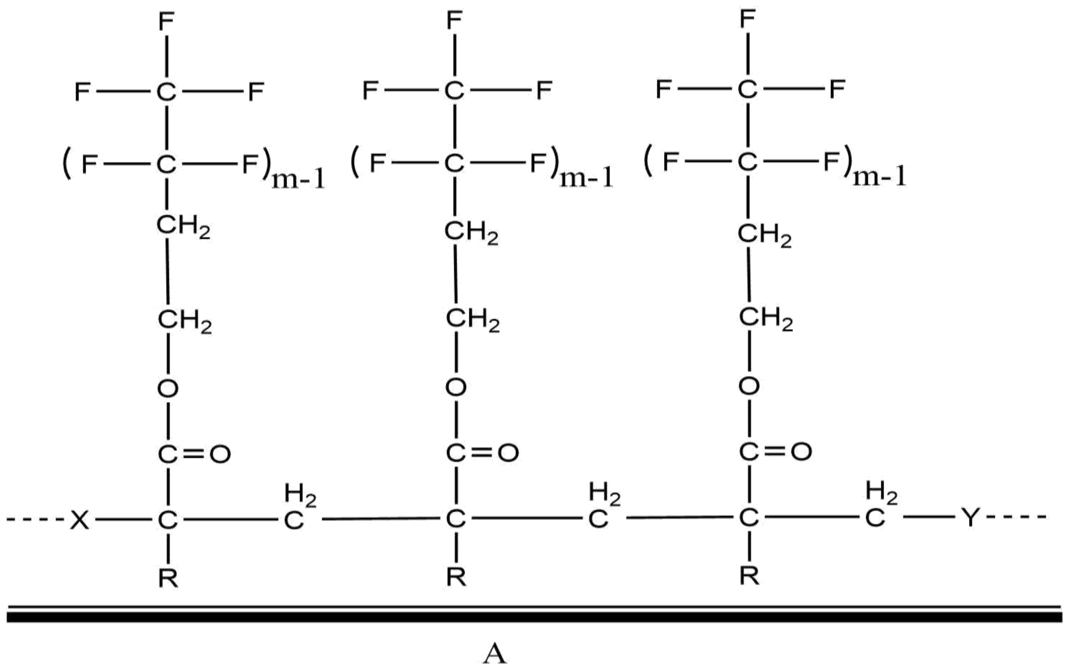


Fig. 1. Fluorocarbon polymer on fibre surface. Where m= **6 – 10**, X and Y are co-polymers mainly stearylacrylates, R= H or CH_3 (polyacrylic or polymethacrylic acid esters), and A is the fire surface.

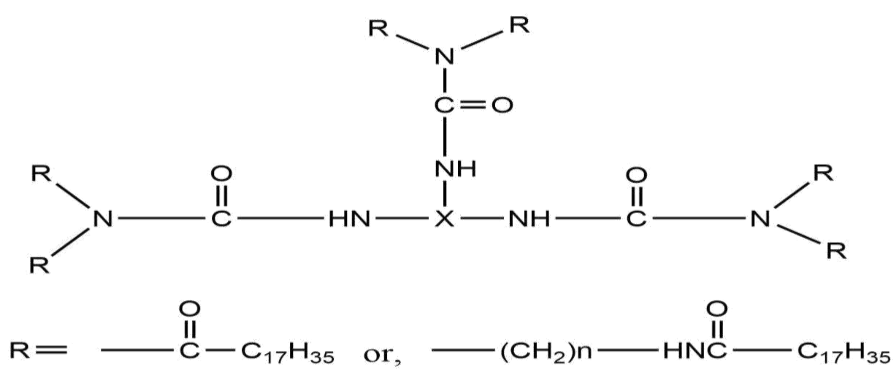


Fig. 2. A dendrimer structure synthesized from three distearyl-amines or amides and a tri-functional isocyanate X (N=C=O).

Experimental

Materials

100% cotton single jersey knitted fabric (Yarn count: 24 Ne carded yarn; Stitch length; 2.92 mm; GSM; 180) was used, which was collected from Fakhruddin Textile Mill Ltd., Sreepur, Gazipur. “RUCOSTAR EEE6” was used as a water- and oil- repellent chemical, which was C_6-FC-dendrimer resin with polymeric hyperbranched dendrimers in a hydrocarbon matrix. Non-ionic **p**olysiloxane (“RUCOFIN HSF”) was used as a softening agent and blocked polyisocyanate (“RUCO LINK-RCX”) was used as a cross-linking agent. All chemicals were purchased from Spectra Dry-Chem (PVT.) Ltd., Germany.

Sample preparation

Five samples of 60, 70**,** 80, 90 and 100 g/L FC-dendrimer resin each plus 15g/L blocked polyisocyanate and 10 g/L polysiloxane compound were prepared. At first, 100% cotton single jersey knitted fabrics were cut with a dimension of 12 inches x 12 inches. The solution was prepared according to recipe and material to liquor ratio of 1:30 was used. The samples were immersed in the solution. After that the samples passed through two nip rollers on a padding machine to squeeze out the excess solution at two bar pressure, leaving the fabric with a more limited amount of the chemical remaining. Then the samples were dried at 100° C. Finally, the samples were cured at 150°C, for two minutes, with the help of a mini-stenter machine. Cotton fabric, untreated fabric, control are used in this study instead of 100% cotton bleached single jersey knitted fabrics. The sample preparation process is shown in **Fig. 3.**

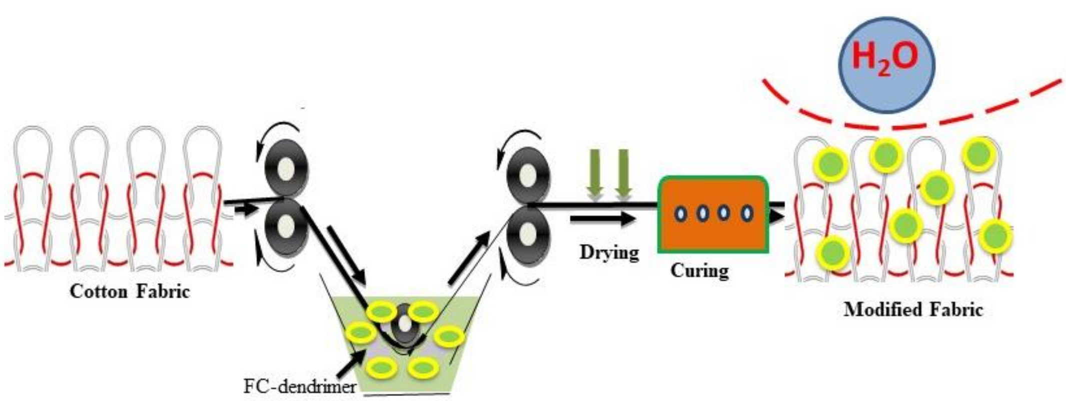


Fig. 3. Schematic diagram of Sample preparation process

Fourier transform infrared spectroscopy (FTIR)

The dried samples were then mixed with KBr in the ratio of 1:100 using mortar and pestle. An FTIR spectrophotometer (Spectrum-100, Perkin Elmer, USA), with a scanning range of 400-4000 cm^{-1} was used to carry out the test.

X-Ray diffraction (XRD) analysis

The crystallinity of the treated and untreated samples was evaluated by X-Ray diffraction (XRD) analysis using X' Pert PRO diffractometer (PAN Analytical, Auckland, New Zealand). The XRD was carried out using Cu-Kα radiation, of wavelength $\lambda = 0.15406$ nm, as the X-ray source. The operating voltage and current were 40kV and 30mA, respectively, at room temperature. The sample holder was placed in the X-ray goniometer and a 2θ scanning range, over 5º–50°, was used.

The 'd' spacing was evaluated according to Bragg's law:

$$d\lambda = 2d\ \mathrm{Sin}\theta \text{ -------------- } (1)$$

where, λ is 0.15406 nm is the X-ray wave length and d is the inter plane spacing.

The full width of the peak at half-maximum (FWHM) value of the XRD pattern was used to estimate the crystal diameter, using the Deby-Scherrer formula **(Jenkins and Snyder 1996):**

$$D = \frac{K\lambda}{\beta \mathrm{Cos}\theta} \text{ .... } (2)$$

In this equation, K (= 0.9) is the Scherrer constant, the X-ray wave length is $\lambda = 0.15406$ nm, θ is the Braggs diffraction angle, and β is the full width of the peak in the X-ray pattern line at half peak-height in radians.

The crystalline percentage (Cr %) of treated and untreated samples was calculated from the XRD pattern, using the following equation **(Ye and Farriol 2005) :**

$$Cr\ \% = \frac{I_{002} - I_{am}}{I_{am}} \times 100 \text{ --------------------------- } (3)$$

Where:

I_{002} is representing the crystalline region of the material, and Iam is representing the amorphous region of the material in cellulose fibres.

Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS)

A scanning electron microscope was used to study the surface morphology of the cotton fabric both before and after modification. SEM (Model- Phenom G2 pro, Netherland) was performed to investigate the surface morphology at × 3000 magnification.

The energy dispersive X-ray (EDS or EDX) represents the different elements composing the materials of the sample. The contents of carbon (C), nitrogen (N), oxygen (O), Fluorine (F) of the treated cotton were measured by EDS (JEOL-6300F, Germany).

Thermogravimetric analysis (TGA)

A simultaneous TGA and DTA thermal analyzer (Jupiter, Germany) studied both the FC-dendrimer treated and untreated samples. The samples were heated to 20°C to 800°C with a heating rate of 10°C per minute in a nitrogen atmosphere. The mass of the sample and its temperature were recorded continuously, to determine the mass lost.

Water repellence test

A spray rating tester (Model no: 333A, Mesdan Lab, Italy) helped to assess the water repellence of the cloth. AATCC Test Method **(2009)** was followed in such assessment. For testing, a piece of cloth of 180 mm in diameter was stretched tightly in embroidery hoops. They were held at a 45^{o} angle. Then 250 mL of water was sprayed on it from 150 mm height. The fabrics were rated between 0 and 100 on the basis of photographic pattern, with the stages of 100, 90, 80, 70, 50, and 0. Zero (“0”) rating revealed complete wetting whereas 100 ratings pointed out completely dry of the upper and lower surfaces.

Drop test

A drop test was also carried out, to observe the repellent properties of the treated fabric against aqueous dye solution and paraffin oil **(Singh and Singh 2016).**

Wash durability test

A wash durability test was carried out to check the water repellency after several times washing. Treated fabrics were washed by industrial washing machines according to AATCC 124-2018 **(2018)** standard**.** In the current job, washing was done just five times.

Water contact angle measurement

Using the goniometer (DSA 100, Kruss, Germany) sessile drop technique, the finished cotton sample was assessed for contact angle measurement with water. 5 μL of water, by volume, was dropped at points on the sample in five places, at room temperature (**Siddiqui et al. 2017).** The average value of contact angles was used. The drops were photographed.

Hydrostatic pressure test

A hydrostatic pressure test was evaluated according to AATCC Test Method 127-2008 **(2009).** The lower surface of the tested fabric was subjected to hydrostatic pressure head, increasing the pressure at a constant rate, until leakage appeared in three points on the upper surface of the test specimen. A reservoir with a circular test area of 100 ±5 cm^2 contains distilled or deionized water which is applied to the fabric surface. Results were obtained from water pressure by a millibar (mb).

Oil repellence test

The ability of the treated fabric to repel oil was tested by the 3M Oil Repellence Test **(Khoddami et al. 2015).** In this test, a drop of oil was dropped on to the sample while it was lying flat on a smooth horizontal surface. The droplets were observed for 30 s from a 45^o angle. Standard test liquids for this test are shown in **Table 1.**

Table 1 Standard test liquids for 3M oil repellency test method

Composition of test liquid	3M oil repellency rating number	Surface[a] tension (mN/m)	Density[a] ((Kg/dm^3)
Paraffin oil	1	31.5	0.86
65/35 paraffin/n-hexadecane	2	29.6	0.82
n-hexadecane	3	27.3	0.77
n-tetradecane	4	26.4	0.76
n-dodecane	5	24.7	0.75
n-decane	6	23.5	0.73
n-octane	7	21.4	0.70
n-heptane	8	19.8	0.69

[a] Data are cited from Handbook of Chemistry and Physics **(Haynes 2017).**

Acid resistance test

The extent to which the fabric was acid-resistant was tested by an acid drop test. 20µL of 98% sulfuric acid was dropped on the fabric with a micropipette. A digital camera then recorded the ensuing damage (**Wang et al. 2018).**

Oil-water separation test

FC-dendrimer coated fabric was used to perform an oil-water separation test, according to the Yang et al**., (Yang et al. 2019)** method with some amendment. For this purpose, a mixture of oil and water, 50 mL hexane and 50 mL red-dyed was poured on FC-dendrimer coated fabric contained in a funnel placed on a conical flask. The filtrate mixed solution was collected in the flask and FC-dendrimer coated fabric separated them. The following formula was used to measure the efficiency of separation:

$$\text{Efficiency} = \frac{V_a}{V_b} \times 100\text{-------------} \quad (4)$$

Where, V_b is the volume of oil before separation and V_a is the volume of after separation.

Self -cleaning (Anti-fouling) tests

Self-cleaning tests, for both untreated cotton and FC-dendrimer polymer-coated cotton, were carried out according to Yang et al.'s, **(Yang et al. 2019)** method, with some modification. Very fine Methylene Blue (MB) dye powder was used as a surrogate for soil. The Methylene Blue powder was placed on untreated cotton and FC-dendrimer-polymer-coated cotton was dissolved in water and flowed with a water stream. After cleaning with water, the sample was dried and takes the image of the samples.

Flame retardant test

The flammability of treated and untreated fabric was evaluated by a 45º flammability tester (Govmark, Model No. TC-45, USA) according to the ASTM D 1230-94 standard (**1994).** On the other hand, limiting oxygen index was carried out according to ASTM D2863.

Measurement of UV protection factor (UPF)

A UV spectrophotometer (Shimadzu 1650, Japan) was used to measure the ultraviolet protection factor and calculated the factor according to the AATCC Test Method: TM 183-2004 (**2010)** by the following equation:

$$\text{UPF} = \frac{\sum_{280}^{400} E_\lambda . S_\lambda . \Delta_\lambda}{\sum_{280}^{400} E_\lambda . S_\lambda . \Delta_\lambda . T_\lambda} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (5)$$

In this equation: E_λ is erythermal spectral effectiveness, S_λ, is solar spectral irradiancies, λ is the wavelength in nm, T_λ is spectral transmission and Δ_λ is wavelength intervals (in nm).

Antibacterial activity test

Disc diffusion process was involved to test an inhibition zone, for both treated and untreated samples. The AATCC Test Method: TM 147-2004 (**2010)** was used.

Fabric weight

Fabric weight per unit area was measured according to the Standard ASTM- D 3776-96 **(33. 2002).**

Fabric thickness

The thickness of the fabric using James Heal's thickness gauge followed B.S. 2544:1954 (**1954).**

Bursting strength

A Trust Burst tester, ASTM (D 3786 87 **(1987)** applied a force to the plane of the fabric at 90^0, under specified conditions, until it ruptured the cloth.

Abrasion Resistance

The abrasion resistance was tested in accordance with ASTM D 4966-98 by using a Martindale Abrasion Tester.

Thermal conductivity

A Lee disc apparatus was used to measure the ability of the samples to conduct heat by the following formula **(Vigneswaran et al. 2009):**

$$\text{Thermal conductivity, } K = \frac{msd \times \frac{d\theta}{dt}}{a(t_1 \quad - t_2)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (6)$$

Where, K is the ability of the of the sample to conduct heat, m is the brass disc mass, s is the specific heat of the disc material, d is the specimen thickness (in mm), $\frac{d\theta}{dt}$ is the cooling rate, a is the area of the specimen cross-section, t_1 is the highest temperature persisting over time and t_2 is the lowest temperature persisting over time.

Air permeability

Air permeability of the fabric was tested by the air flow method **(BS 5636, 1990).**

Water vapor permeability

The BS 7209 test **(1990)** was used to measure the water vapor permeability of the sample using the following formula:

$$\text{Water vapor permeability} = \frac{24\,M}{At}\ \text{gm/m}^2\text{/day} \quad \text{---------------------} \quad (7)$$

In the above equation, M is mass loss (in grams), A is the sample's exposed area in m^2 and t is the time elapsed between weighing's of the assembly (in hours)

Statistical analysis

Statistical analysis was used to investigate the acceptable level of thermal comfort properties. For this purpose, the Chi-square distribution Test Method was carried out at a 5% level of significance **(Gupta 2002)**

Results and discussion

The manuscript has been organized so that the first confirmation tests, such as FTIR, XRD, EDS, SEM, and TGA, are discussed. Second, functional properties such as water and oil repellent, oil-water separation, self-cleaning, flame retardant, antibacterial, and UV protection have been addressed. Physical and mechanical properties have been addressed in the third section. Fourth, thermal conductivity, air permeability, and water vapor permeability have all been explored as comfort properties. Finally, the comfort data were analyzed statistically. Each result will be analyzed and discussed below.

FTIR spectra

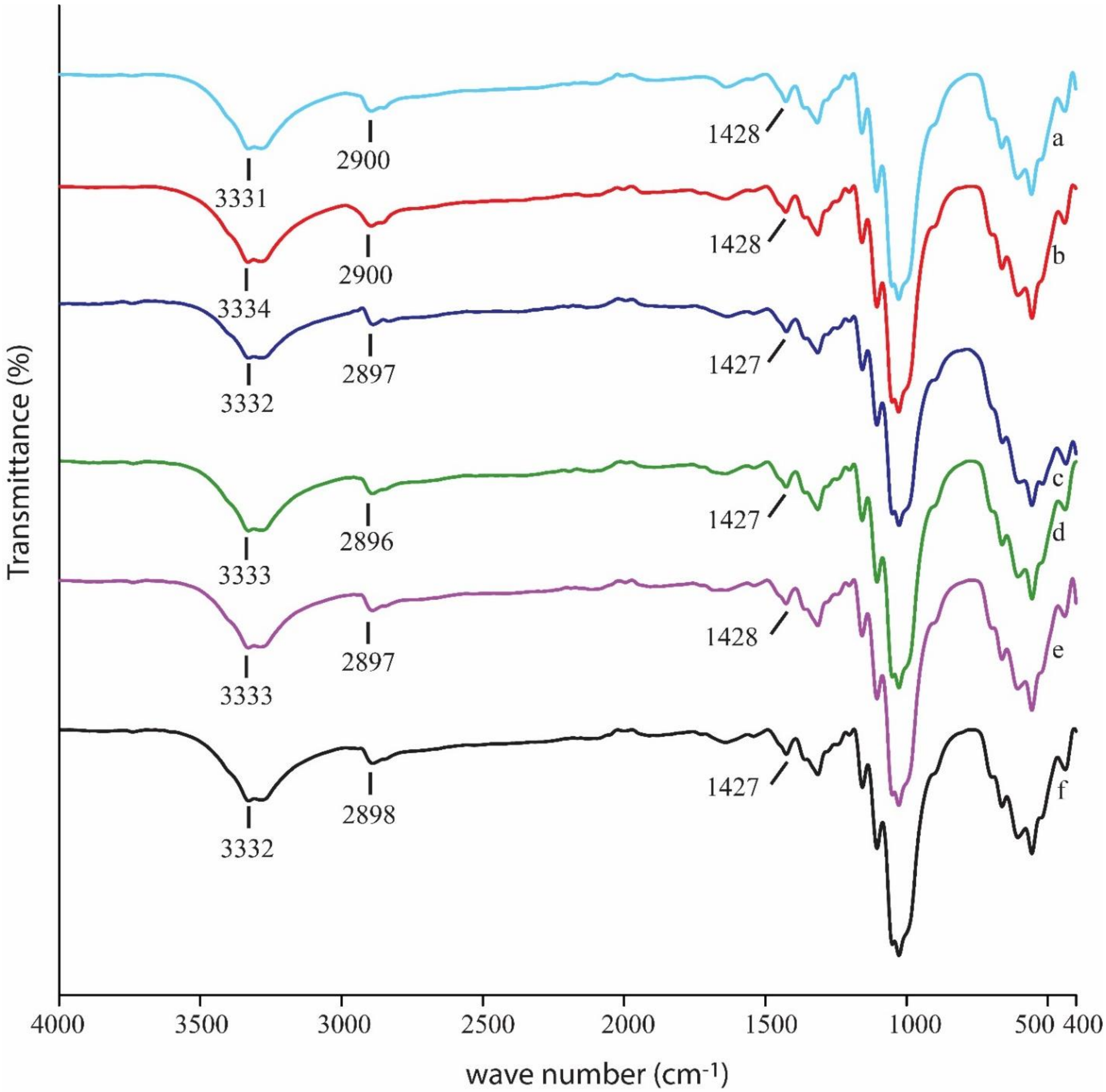


Fig. 4. FTIR spectra of (a) Untreated fabric, (b) 60 g/L FC-dendrimer, (c) 70 g/L FC-dendrimer, (d) 80 g/L FC-dendrimer, (e) 90 g/L FC-dendrimer and (f) 100 g/L FC-dendrimer treated fabric.

In **Fig. 4a,** FTIR spectra of scoured bleached cotton show a broad peak at 3331 cm^{-1}. This may signify OH stretching vibration of cellulose hydroxyl groups in the region of 3100-3600 cm^{-1}. The spectra of the scoured bleached cotton fabric also showed typical characteristic peaks at 1428 $cm^{-1}$and 2900 cm^{-1}, which may be C-H bending and stretching bands, respectively. **(Chung et al. 2004).**

Wavenumber 3331 cm^{-1} of bleached cotton fabric has been converted into little bit high wave numbers of FC-dendrimer treated cotton fabrics as shown in **Fig. 4(b, c, d, e, f),** which indicated intermolecular hydrogen bond formation. The peak at 2900 cm^{-1} of bleached cotton fabric has been shifted to a little bit lower wavenumbers of the FC-Dendrimer treated fabrics except 60 g/L. A peak at 1427 cm^{-1} has been also changed after FC-dendrimer treatment. These changes prove that FC dendrimer successfully attached to the cotton fabric.

XRD Analysis

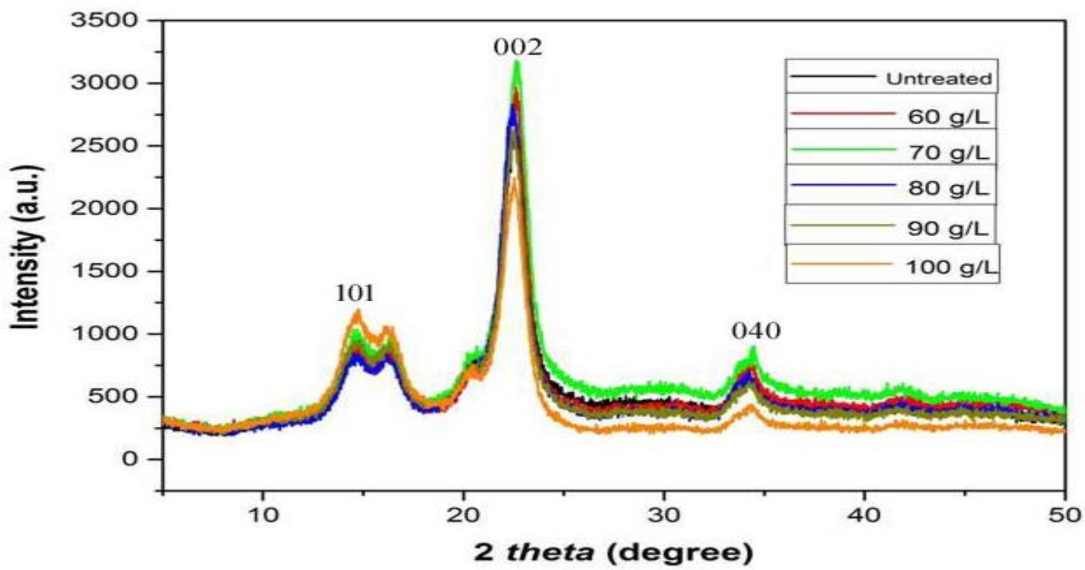


Fig. 5. X-Ray Diffraction pattern untreated and FC-dendrimer treated fabrics.

Table 2 Measurement of crystallinity percentage (Cr %) of untreated and FC-dendrimer treated fabrics.

Observation	2 theta (°) in the CR	Intensity in counts	2 theta (°) in the AR	Intensity in counts	Cr%
60 g/L FC-dendrimer	22.558	2333	18	433	81.44
70 g/L FC-dendrimer	22.599	2413	18	483	79.98
80 g/L FC-dendrimer	22.434	2246	18	432	80.76
90 g/L FC-dendrimer	22.462	2028	18	409	79.83
100 g/L FC-dendrimer	22.441	2166	18	478	77.93
Untreated fabric	22.506	2519	18	448	82.21

N.B: Crystalline Region (CR), Amorphous Region (AR)

Table 3 Measurement of crystal diameter and inter plane spacing (d) of untreated and FC-dendrimer treated fabrics.

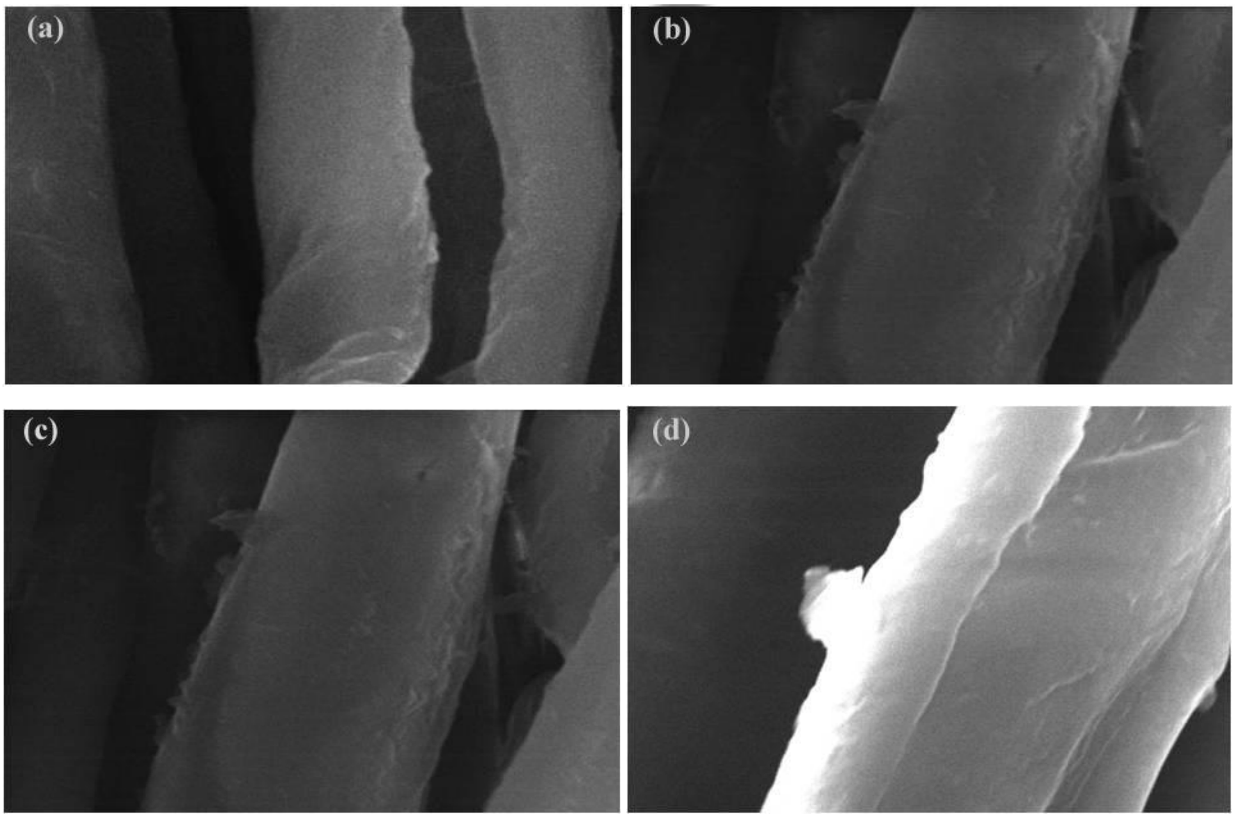
Observation	2 theta (°) in the CR	Inter plane spacing 'd' in nm	FWHM (°)	Crystal diameter (nm)
60 g/L FC-dendrimer treated	22.558	0.3938	1.40631	5.77
70 g/L FC-dendrimer treated	22.599	0.3932	1.49682	5.44
80 g/L FC-dendrimer treated	22.434	0.3960	1.61561	5.04
90 g/L FC-dendrimer treated	22.462	0.3954	1.56089	5.17
100 g/L FC-dendrimer treated	22.441	0.3958	1.68771	4.89
Untreated fabric	22.506	0.3949	1.37461	5.84

* FWHM- Full width at half maximum

The untreated and FC-dendrimer-treated fabrics both showed three distinctive peaks, at the crystal planes of (101), (002) and (040) as shown in **Fig. 5**. This result agrees with the result found by Altınışık et al., **(Altınışık et al., 2013)** for cellulosic fibre. The crystallinity percentages of 60g/L, 70g/L, 80g/L, 90g/L, and 100g/L FC-dendrimer-treated samples were 81.44, 79.98, 80.76, 79.83, and 77.931, respectively. The crystallinity percentage of the treated samples was slightly decreased compared to those of the untreated samples (82.21%), as the peak intensity of the treated fabric was also decreased when 2θ value at the crystal plane was (002). Crystallinity percentage of the FC-dendrimer-treated samples gradually decreased as the FC concentration increased. It is assumed that the FC polymer created intermolecular hydrogen bonding in the crystalline region of the cotton fabric, implying that some crystalline bonds may have been broken. As a result, the FC-dendrimer-treated fabric exhibited a slightly lower level of crystallinity. On the other hand, the full width at half maximum (FWHM) value of the FC-dendrimer-treated sample was greater than the untreated sample. The full width at half maximum (FWHM) value is inversely proportional to the crystal diameter. This is inherent in the Deby-Scherrer formula (equation no. 2). When full length at half maximum value increases, the crystal diameter of the sample decreases. Thus, the crystal diameter of all FC-dendrimer-treated cotton fabrics was lower than that of the untreated fabrics. A higher crystal diameter implies a higher crystallinity percentage. A positive correlation was clear between crystallite diameter and crystallinity percentage.

No major difference is seen in the inter-plane spacing of untreated and FC-dendrimer-treated fabric as shown in **Table 3.** The X-ray diffraction shows that the characteristic peaks of untreated and FC-dendrimer-treated fabric are almost the same. Thus, the cotton fibre cellulose did not produce any remarkable changes after FC-dendrimer incorporation.

Scanning electron microscopy (SEM) study



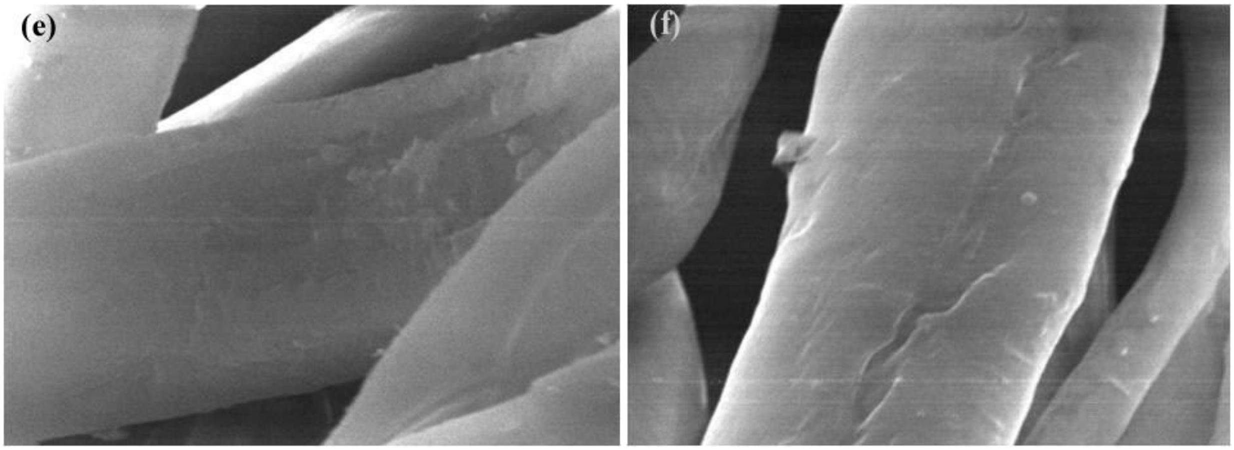


Fig. 6. SEM photograph at $\times$ 3000 magnification and $1\mu m$ scale of (a) Untreated fabric, (b) 60 g/L FC-dendrimer, (c) 70 g/L FC-dendrimer, (d) 80 g/L FC-dendrimer, (e) 90 g/L FC-dendrimer and (f) 100 g/L FC-dendrimer treated fabric.

The surfaces of both the treated and untreated fabrics were studied by scanning electron microscope. The untreated fabric has a smooth surface, as shown in **Fig. 6(a).** The treated fabrics had rough and uneven surfaces, as shown in **Fig. 6 (b, c, d, e, f)** compared to that of untreated fabric. Granular material was clearly visible in **Fig. 6e.** That is what gave the treated fabric its uneven surface and also provides evidence that the water repellent agent was successfully attached to the untreated fabric surface. After FC-dendrimer treatment, the surface texture of the cotton fabric became similar to that of non-wettable fabrics. Among the samples, the uneven appearance of the surface of those treated with 90 g/L and 100g/L concentrations of FC-dendrimer treatment was most prominent. This result is similar to the outcome of Jeyasubramanian et al. **(Jeyasubramanian et al. 2016).**

EDS Analysis

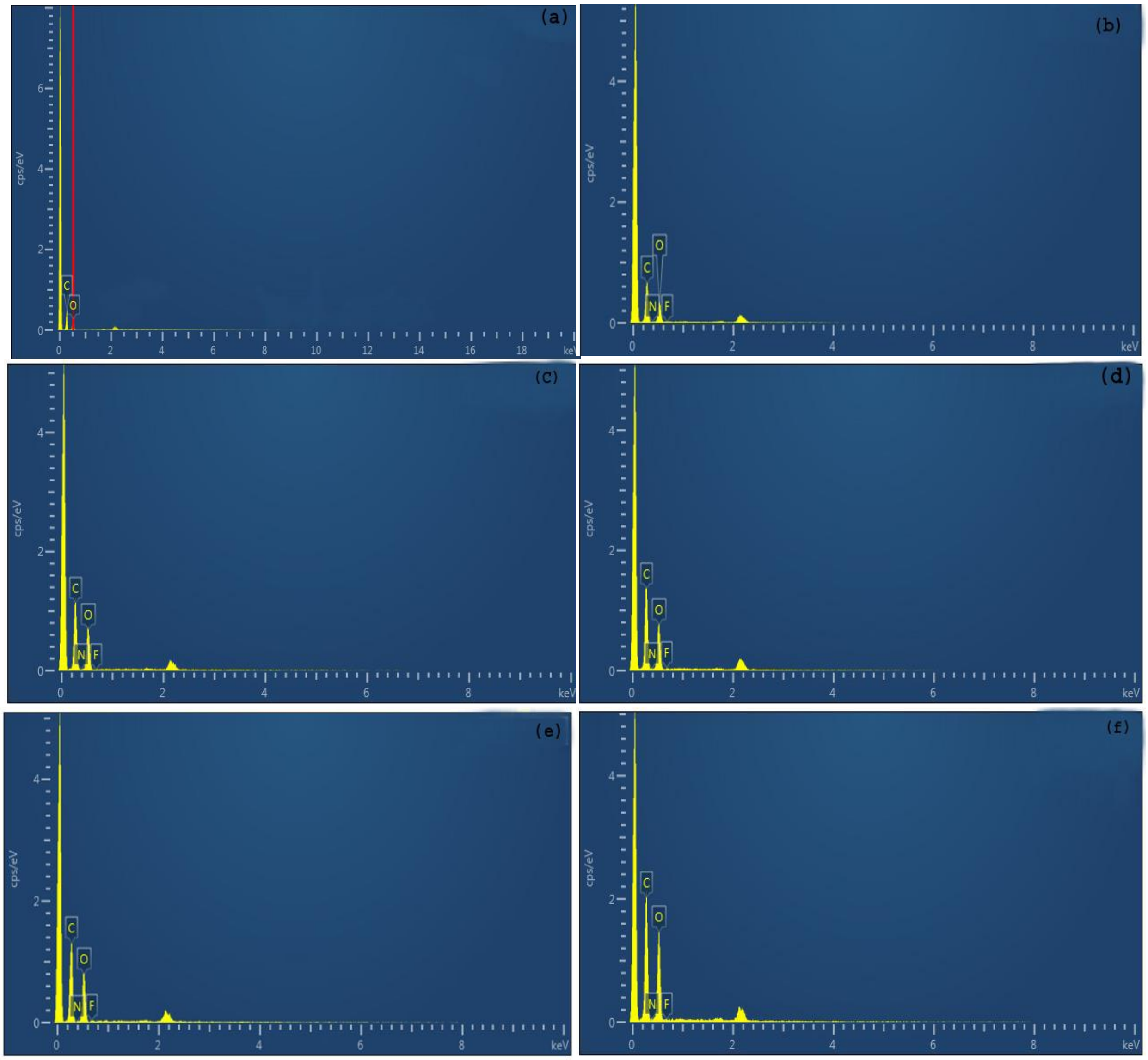


Fig. 7. EDS photograph of (a) Untreated fabric, b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

Table 4 Weight % of untreated and treated samples of EDS spectra.

Elements	Untreated fabric	60 g/L treated fabric (Wt.%)	70 g/L treated fabric (Wt.%)	80 g/L treated fabric (Wt.%)	90 g/L treated fabric (Wt.%)	100 g/L treated fabric (Wt.%)
C	49.85	52.15	53.17	53.53	55.23	56.08
N	00	0.20	0.27	0.38	0.57	1.63
O	50.15	47.53	45.97	45.14	43.09	41.97
F	00	0.12	0.33	0.60	0.82	1.43

The weight percentage of carbon atoms were 49.85, 52.15, 53.44, 53.91, 55.80, 56.08% in untreated cotton and 60 g/L, 70 g/L, 80 g/L, 90 g/L and 100 g/L FC-dendrimer-treated cotton fabrics, respectively, as shown in **Table 4.** The weight percentage of oxygen atoms were 50.15, 47.53, 45.97, 45.14, 43.09, and 41.97% in untreated cotton, and 60 g/L, 70 g/L, 80 g/L, 90 g/L and 100g/L in FC-treated cotton fabrics, respectively. The weight percentage of oxygen atoms in FC-dendrimer-treated fabric was less than that in untreated fabric. Less weight of oxygen peaks in FC-dendrimer-treated fabric indicates that less hydroxyl groups (OH), which attract water molecules¸ were present. But the weight percentage of carbon atoms was greater in the treated fabric. The greater weight for carbon peaks in FC-dendrimer-treated fabric implies that the number of hydrocarbon groups has increased. This, in turn, implies that hydrophobicity increased with increasing the concentration of fluorocarbon polymer.

Thermal analysis

Table 5 TGA and DTA thermograph data of treated and untreated fabrics.

Observation	Weight loss % at 100°C	Nature of DTA peak	DTA 1st Peak Temp. (°C)	Weight loss % at DTA 1st Peak Temp. (°C)	DTA 2nd Peak Temp. (°C)	Char yield % at DTA 2nd Peak Temp. (°C)
Untreated fabric	6	Exothermic	377	82.4	478	2.6
60 g/L FC-dendrimer	5	Exothermic	379	99.5	504	2.3
70 g/L FC-dendrimer	5	Exothermic	376	82.7	506	1.5
80 g/L FC-dendrimer	3	Exothermic	380	82.6	503	1.9
90 g/L FC-dendrimer	4	Exothermic	382	83.2	497	2.6
100 g/L FC-dendrimer	6	Exothermic	382	75.1	495	3.2

Thermal behavior of untreated fabric and FC-dendrimer-treated fabric were evaluated by the study of TGA and DTA thermograms as shown in **Table 5**. The TGA thermogram represents the weight loss percentage of the specimen and shows three stages of thermal degradation. In the first stage, weight loss occurred of untreated, 60, 70, 80, 90 and 100 g/L FC-dendrimer treated fabrics was 6, 5, 5, 3, 4 and 6% respectively at 100°C due to loss of moisture, low molecular weight solvent and gas. Major weight losses were observed in the second stage, at DTA 1st peak temp. as shown in **Table 5.** The DTA 1st peak temperature of the untreated fabric, 60, 70, 80, 90 and 100 g/L FC-dendrimer-treated fabrics was 377, 379, 376, 380, 382, 382°C respectively and a more pronounced degradation process occurs at this temperature due to break down of the glycosidic linkages and hydrogen bonds leading to the formation of organic compounds. Final stage residue char is obtained at DTA 2nd peak temperature (**Table 5**). From the **Table 5**, it is clear that the thermal stability of FC-dendrimer-treated fabric is slightly increased.

Spray test results

Table 6 Standard Spray test rating chart according to AATCC -22 method **(Khoddami et al. 2015**).

AATCC-22 Standard rating	Note
AATCC 100	100: No sticking or wetting of upper surface
AATCC 90	90: Slight random sticking or wetting of upper surface
AATCC 80	80: Wetting of upper surface at spray points
AATCC 70	70: Partial wetting of whole of upper surface
AATCC 50	50: Complete wetting of whole surface
00	0: Complete wetting of whole upper and lower surface

Table 7 Spray test readings of FC-dendrimer resin treated fabric

Observation	Spray Test Readings (AATCC-22)	After five times laundering (AATCC-22)
60 g/L FC-dendrimer treated	80	80
70 g/L FC-dendrimer treated	90	90
80 g/L FC-dendrimer treated	90	90
90 g/L FC-dendrimer treated	100	100
100 g/L FC-dendrimer treated	100	100

Table 7 shows spray-test results for FC-dendrimer-treated fabric. The spray test reading, according to AATCC-22, of 60, 70, 80, 90 and 100 g/L FC-dendrimer-treated samples were 80, 90, 90, 100 and 100, respectively. According to the rating chart, 90 g/L and 100g/L show the highest rating. This rating implies that there was "No sticking or wetting of the upper surface". When the concentration of FC-dendrimer was 90 g/L and 100 g/L, the weight gain percentage was maximized. As a result, the thickness of the treated fabric also increased. So, the treated fabric showed excellent water resistance performance. Again, at 70 g/L and 80 g/L, the treated sample

had a rating of “90’’ meaning “Slight random sticking or wetting”. After five times launderings, the spray rating value for all five samples remained unchanged. Repellent finishes achieve repellence because they reduce the free energy at the fibres’ surface. A drop of liquid will spread, if, the adhesive interactions, between the fibres and the liquid drop, are greater than the internal cohesive interactions within the liquid. A drop of liquid will not spread, if, the adhesive interactions, between the fibres and the liquid drop, are lower than the internal cohesive interactions within the liquid. Surfaces that exhibit low interaction with liquids are referred to as “low-surface energy” **(Schindler and Hauser 2004).**

Water contact angle of the samples

Contact angle measurement is the best method to evaluate the water repellence of a hydrophobic surface. The static contact angle is the angle between a liquid and the surface which is created when the liquid contacts the surface. If the water contact angle is $0\leq \theta \leq 90°$, the surface is defined as “hydrophilic”. If the water contact angle is $90\leq \theta \leq 180°$, the surface is defined as “hydrophobic”. Water repellency increased when cotton fabric with low surface energy has obtained.

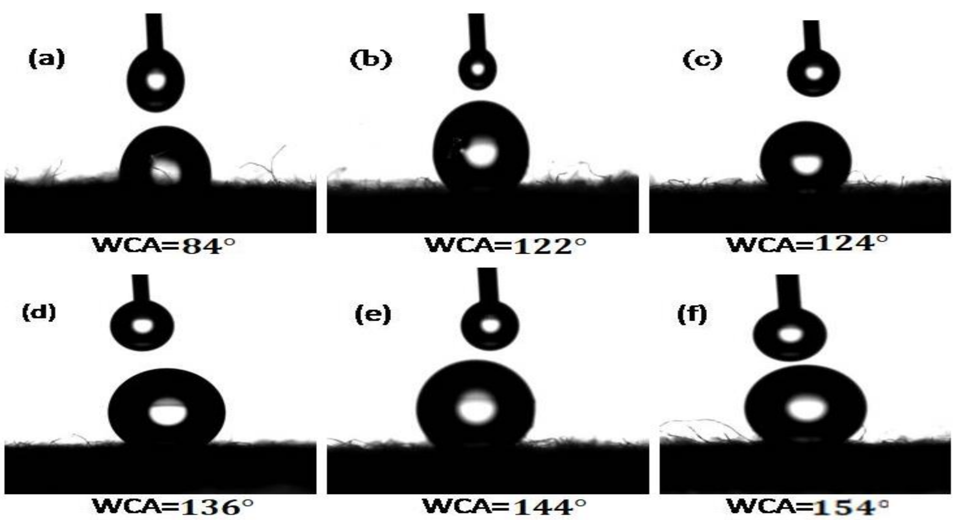


Fig. 8. Water contact angle (WCA) of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

Water contact angle indicates whether a surface is hydrophilic or hydrophobic because it measures how much spread a drop of liquid on the surface. If oxidizing agents or ion-able groups are introduced onto the cotton surface, the surface will become more hydrophilic. As a result, a water droplet will spread across the surface easily and the water contract angle will become smaller. On the other hand, if hydrocarbons are introduced onto the surface, the surface will become more hydrophobic and the water contract angle will become larger. Therefore, to measure the water contact angle, a droplet of water is placed on the surface. The height and width of the water's spread are then recorded to calculate the angle.

The 100 g/L and 90 g/L FC-dendrimer treated cotton fabric provided excellent water repellence: its water contact angle measured between 154° and 144°, as shown in **Fig. 8 (e, f).** 60 g/L, 70 g/L and 80 g/L FC-dendrimer-treated cotton fabrics exhibited water contact angles of 124°, 122° and 136°, respectively, as shown in **Fig. 8 (b, c, d).**

Drop test results

In this study, 100 g/L FC-dendrimer resin plus 15 g/L blocked polyisocyanate and 10 g/L polysiloxane compound were used to prepare the sample. A colored water droplet and paraffin oil are spread easily on an untreated fabric surface in **Fig. 9(a) & 5(b)** respectively. On the other hand, colored water droplets and paraffin oil did not spread on the FC-dendrimer treated fabric surface in **Fig. 9(c).** The spherical shape of the water and oil droplet is shown in **Fig. 9(c).** Thus, FC dendrimer-treated cotton fabrics are water and oil repellent.

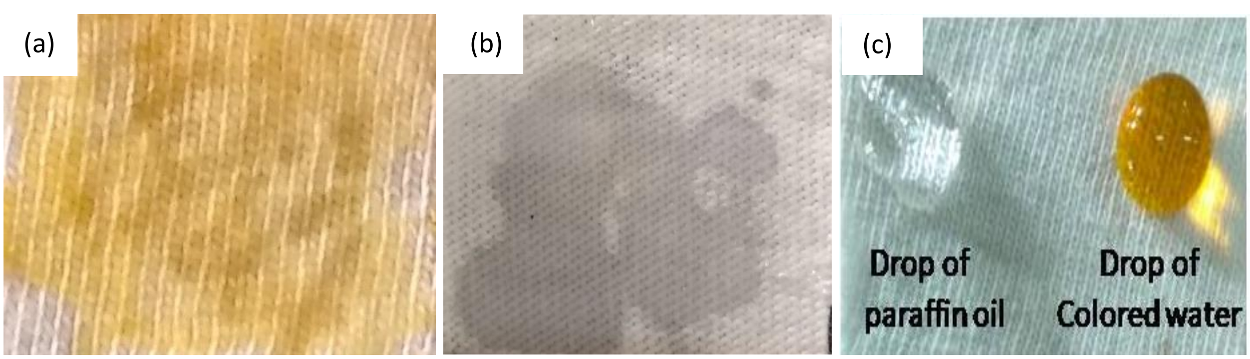


Fig. 9. Water-oil repellent properties of (a) & (b) Untreated fabric and (c) 100 g/L FC-dendrimer treated fabric.

Hydrostatic pressure test result

Table 8 hydrostatic pressure test result

Observation	Hydrostatic Pressure (mbar)
Untreated fabric	140
60 g/L FC treated fabric	158
70 g/L FC treated fabric	162
80 g/L FC treated fabric	168
90 g/L FC treated fabric	175
100 g/L FC treated fabric	180

Hydrostatic pressure means the pressure needed for water to penetrate a fabric. Hydrostatic pressure increased after FC-dendrimer coating, compared to that of untreated fabric. **Table 8** shows this increase. Hydrostatic pressure increased gradually with the increase of coating because the FC-dendrimer polymer coating on the fabric becomes denser as the amount of chemicals is increased. A more dense coating makes the fibre more water-repellent. The highest hydrostatic pressure (180 mbar) and lowest hydrostatic pressure (158 mbar) were reached with coatings of 100 g/L and 60 g/L treated fabrics, respectively. Hydrostatic pressure is directly proportional to the water repellency property. High hydrostatic pressure indicates the high repellent property. So it can be said that 100 g/L FC-dendrimer finished fabric exhibits the highest water repellent property.

Oil repellence

The surface tension and density of paraffin oil are 31.5 mN/m and 0.84 kg/dm^3, respectively, as shown in **Table 1.** Kasturiya & Bhargava **(Kasturiya and Bhargava 2003)** found that the critical surface tension for FC-dendrimer polymer-coated surfaces is 6 mN/m - 28 mN/m. Yet, for bleached cotton, this critical surface tension is 44 mN/m. When FC-dendrimer polymer is applied to bleached cotton fabric, the surface tension is reduced to 6 mN/m - 28 mN/m. Paraffin oil did not penetrate the FC-dendrimer-treated fabric, as the surface tension of paraffin oil is greater than that of the FC-dendrimer-treated fabric. On the other hand, paraffin oil easily penetrated the bleached cotton fabric, since the surface tension of paraffin oil is less than that of the bleached cotton fabric. So it can be said that the 60 g/L, 70 g/L, 80 g/L, 90 g/L and 100 g/L FC-dendrimer treated fabric showed paraffin oil repellence.

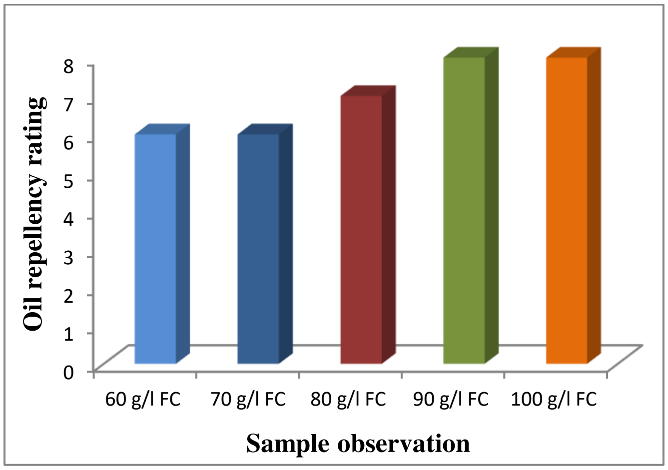


Fig. 10. Oil repellency rating of the FC-dendrimer treated sample.

The surface tension and density of n-heptene are 19.8 mN/m and 0.69 kg/dm^3, respectively. The surface tension of n-heptane is less than that of the other organic oils which were used in the oil repellence test, as shown in **Table 1**. N-heptane organic oil can easily wet 60 g/L, 70 g/L, and 80 g/L FC-dendrimer-treated fabric, but not 90 g/L and 100 g/L FC-dendrimer-treated fabric. Therefore, 90 g/L and 100 g/L FC-dendrimer-treated fabric showed an oil repellence rating of 8 (eight), according to the oil repellence test chart, as shown in **Table 1**. The high value of water contact angle also indicated the high degree of oleophobicity. The water contact angle of 90 and 100g/L FC- dendrimer-treated fabrics are 154° and 144° as in **Fig. 10 (e, f).**

The surface tension and density of n-octane are 21.4 mN/m and 0.70 kg/dm^3, respectively. n- Octane organic oil can easily wet 60g/L and 70 g/L FC-dendrimer-treated fabric but not 80 g/L FC-dendrimer-treated fabric. So 80 g/L FC-dendrimer-treated fabric achieved the oil repellence rating of 7 (seven), according to **Table 1.**

N-decane organic oil cannot wet the 60 g/L and 70 g/L FC-dendrimer-treated fabric, as the surface tension of n-decane is higher than that of n-octane. So 60 g/L and 70 g/L FC-dendrimer-treated fabric achieved the oil repellence rating of 6 (six) according to **Table 1.** The oil repellence rating of all the FC-dendrimer-treated samples is illustrated in **Fig. 10.**

Oil-water separation

At present oil-water separation becomes a crucial issue due to the generation of considerable oily wastewater from agricultural and industrial activities. On the other hand, improper disposal of used motor oil, oil spills, or leaks from ships or tankers have considerably contaminated water and our food chain. **(Wang et al. 2016; Cao and Cheng 2018; Yuan et al. 2018; Baig et al. 2019).**

All concentrated FC-dendrimer-treated fabrics exhibited hydrophobic characteristics because the surface tension of water is fixed. On the other hand, FC-dendrimer-treated fabric showed oleophobic and oleophilic characteristics which were determined by different surface tensions of the different organic oils tested. For example, n-decane cannot penetrate the 60g/L and 70 g/L FC-dendrimer-treated fabric but n-octane can do so easily. As a result, a mixture of n-octane and water can be separated by 60 g/L and 70 g/L FC-dendrimer-treated fabric. Similarly, a mixture of n- heptane and water has been separated by 60, 70 and 80 g/L FC-dendrimer-treated fabric. In addition, all the FC-dendrimer-treated fabrics can be used to separate the mixture of n-hexane and water, as the surface tension (18.43 mN/m) of n-hexane organic oil is less than that of all the FC-dendrimer-treated fabric.

In this study, 50 mL water and 50 mL n-hexane are mixed, and 100 g/L FC-dendrimer-treated fabric is used for separation. After separation, the collected water remaining is still about 50 mL. The collected oil decreases a little in volume due to the absorbance of cotton and volatilization. The coated cotton would let the oil permeate itself until saturation in the parts which make contact with it. The FC-dendrimer-treated cotton, however, showed excellent oil-water separation. Its separation efficiency was 97-99%, calculated according to **equation (4).** A schematic diagram of oil-water separation is shown in **Fig. 11.**

Fig. 11 Schematic diagram of oil-water separation process.

Materials which can repel water even amidst oily pollutants are very useful in seawater. Thus, coated fabric, as described here, with dual functionality, is a promising material for anti-wetting, self-cleaning, support for aquatic floating devices and as a filtration material for rapid, continuous oil-water separation.

Acid resistance

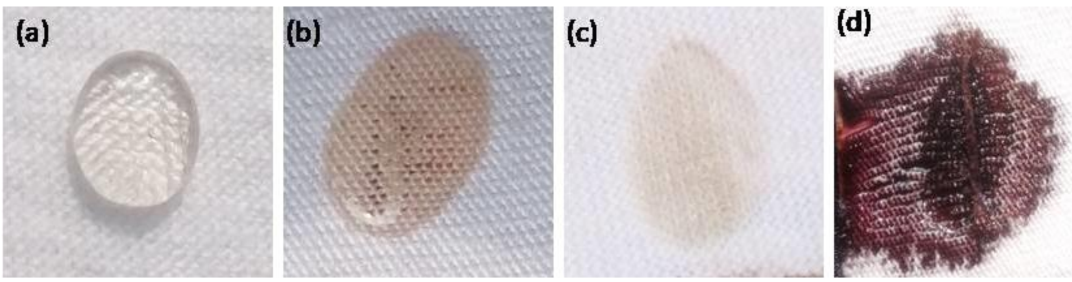


Fig. 12. Acid resistance performances of (a) 100 g/L FC-dendrimer treated fabric after ~~one~~ 1 min., (b) Untreated fabric after ~~one~~ 1 min., (c) 100 g/L FC-dendrimer treated fabric after 30 min. and (d) untreated fabric after 30 min.

Among the possible applications of acid-resistant fabric are petroleum, chemistry, metallurgy and electroplating. Workers in these industries need clothing made of such fabric to protect them from the dangerous acids with which they work. In the past, clothing was made acid-resistant by coating it with rubber or hydrocarbon resins. These resins created a continuous film on the textile which closed its empty spaces, stopping the intrusion of acid **(Forsberg et al. 2014).** Gal'braikh **(2005)** proved that fluoropolymers effectively resist corrosive solutions, including acid. Wang et al. **(2011)** and Zhou et al. **(2013)** used fluorinated polymers which resisted acid most effectively.

Acid resistance performance of the untreated fabric and 100 g/L FC-dendrimer treated fabrics is shown in **Fig. 12**. Initially, 100 g/L FC-dendrimer was not affected by acid droplet **Fig. 12a.** But the untreated fabric was affected by acid droplet to some extent **Fig. 12b**. After 30 minutes, the treated fabric was washed with water. Colour change had taken place where the acid droplet had landed **Fig. 12c**. A large area of untreated fabric was burnt **Fig. 12d** by the acid droplet, after 30 minutes.

Self-cleaning

Water-resistant finishes are chemical additions to clothing that enhance the hydrophobic nature of a fabric's surface. Completely hydrophobic surfaces also must be self-cleaning. Hydrophobic and self-cleaning attributes are exhibited in many surfaces of nature such as the wings of butterflies, the leaves of cabbage and lotus, the elephant's ears and Indian cress. The surface of lotus leaves has protruding nubs, as proven by Scanning Electron Microscopy. Epicuticle wax crystalloid encloses each nub. Due to these crystalloids, lotus leaves clean themselves and have a water contact angle (WCA) of 160. From this model, researchers have created artificial surfaces with water contact angles greater than 90^{o} (**Guo et al. 2011; Lin et al. 2011; Li et al. 2014; Xu et al. 2010).**

Self-cleaning coatings can be:

(1) photocatalysis-induced superhydrophilic coatings and

(2) superhydrophobic coatings.

In super hydrophilic coatings, the surface is cleaned by the sheeting effect of water. Complex organic substances on the surface are broken down into carbon dioxide and water. Superhydrophobic coatings have air pockets that get trapped between nano-structured substrate and water droplets. This results in the formation of a composite solid/air/liquid interface, which leads to an increase in the Contact angle (CA) of liquid droplets. Increased contact angle leads to de-wetting of the surface. Then the droplet rolls off easily, taking away dirt and pollutants with it.

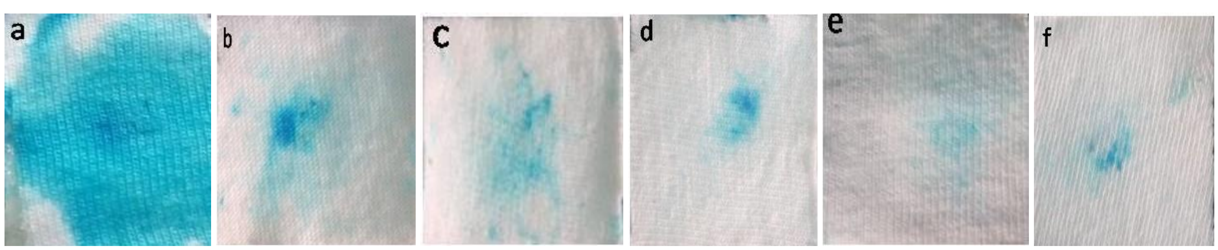


Fig. 13. Self-cleaning performances of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

Fig. 13(a) shows that methylene blue dye powder spread widely on the untreated fabric surface due to the capillary effects of cotton fibers. The coated cotton surface has extremely little adhesion to methylene blue dye powder. However, dirt like methylene blue dye powder on the superhydrophobic cotton surface was quickly and easily removed by water droplets. Thus, the surface in **Fig. 13(b-f)** is clean and dry. Among the treated samples, 80 g/L, 90 g/L and 100 g/L FC-treated samples showed better self-cleaning performance than did the 60 g/L and 70 g/L FC-treated samples.

Flame retardant property

Fig. 14 illustrates that burning times for untreated, 60, 70, 80, 90, and 100 g/L treated fabrics were 10.5, 10.94, 11.44, 13.73, 14.3 and 14.6 seconds, respectively. Burning times of all the FC-dendrimer-treated samples were greater than that for the untreated samples. Burning time gradually increased with an increase in the FC-dendrimer concentration. Burning time mainly depends on the amount of polymer loading on the sample and the amount of oxygen in the sample. Less amount oxygen in the sample increases the burning time. EDS results show that oxygen amount of untreated, 60, 70, 80, 90, and 100 g/L treated fabrics are 50.15, 47.53, 45.97, 45.14, 43.09, 41.97. EDS result clearly indicates that the oxygen amount of untreated fabric was more than treated fabric. These results also revealed that wt% of oxygen decreased with increasing concentrations of FC-dendrimer polymer solution.

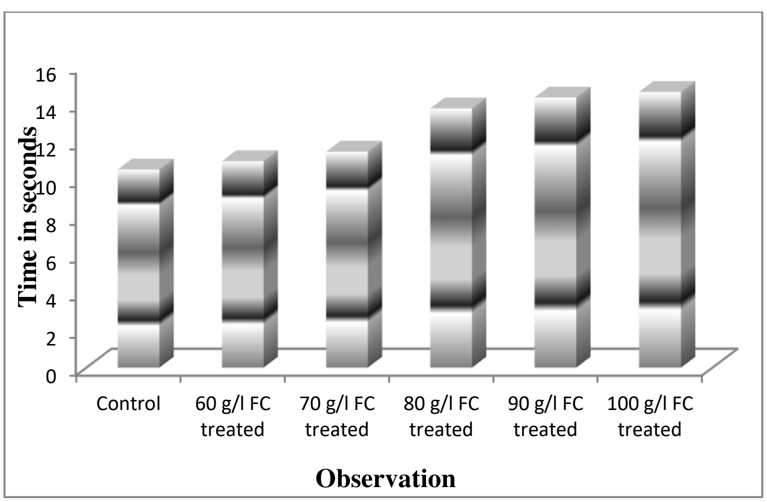


Fig. 14 Burning required time of untreated and treated fabric.

The limiting oxygen index (LOI) is the minimum oxygen concentration needed to support flaming combustion of a substance in a mixture of oxygen and nitrogen. The limiting oxygen index (LOI) of untreated cotton fabric was 17.8, according to the results of a survey. The treated fabric's LOI was 18 or a little higher. As a result, it is possible to conclude that FC-dendrimer treated fabric has a minor flame retardant property that needs to be further investigated.

UV Protection factor (UPF)

The United States Environmental Protection Agency has stated that 80% of the UV rays of the sun reach the Earth, damaging human skin (The United States Environmental Protection Agency 2004), even in cold climates (Skin Cancer Foundation 2017). For this reason, it is essential to wear UV protective garments on cloudy or rainy days as well as sunny days. In recent times, people have been searching for garments that are UV resistant

.There are three kinds of UV light:

(1) UV-A (315-400 nm),
(2) UV-B (280-315 nm) and
(3) UV-C (100-290nm).

UV-C radiation is absorbed by the ozone layer. UV-A and UV-B radiation reach the earth's surface. These are the ones that can cause serious health problems. Ultraviolet radiation (UV-R) on earth is comprised of UV-A and UV-B. Its wavelength range is 280-400 nm.

Clothing is the interface between the environment and the human body. It can reflect, absorb or scatter solar waves. Thus, this ordinary clothing is usually not enough to protect the human body from the harmful effects of UV radiation. For this reason, UV-resistant finishing on textiles is necessary.

Table 9 Ultra-violet Protection Factor (UPF) of untreated and FC-dendrimer treated fabrics.

Observation	Mean UV-A transmission	UPF rating of UV-A	Mean UV-B transmission	UPF rating of UV-B	Mean UV-R transmission	UPF rating of UV-R
60 g/L	0.657	2.16	0.113	4.00	0.476	2.84
70 g/L	0.426	3.16	0.111	4.11	0.315	3.25
80 g/L	0.415	3.21	0.109	4.23	0.314	3.49
90 g/L	0.278	4.22	0.089	4.95	0.221	3.66
100 g/L	0.275	4.58	0.081	5.42	0.213	4.42

UPF value of untreated sample was 0.25 for UV-R

Table 9 illustrates that UPF value gradually increased with increasing FC-dendrimer concentration. The relationship between UPF rating and transmittance value is inversely proportional. The highest UPF rating was found for UV-B (280-315 nm). The UPF value of 100 g/L FC-dendrimer-treated sample was 4.42 for UV-R, which was nine times greater than that of the untreated sample. Pande and Crooks (**Pande and Crooks 2011)** identified the absorbance peak at 280 to 285 nm which, they said, is caused by the dendrimer structure. So, it can be said that dendrimer is the key component for the satisfactory Ultra-violet Protection property of FC-dendrimer-treated clothing.

Antimicrobial activity

Clothing that fights germs is better for human health. Sometimes, as in a medical environment, it is essential. If clothing is water, oil and dirt repellant, as well as UV-protective and antimicrobial, this is almost ideal in many applications **(Attia et al. 2017).**

Antimicrobial clothing materials can be active or passive. Passive materials do not contain bioactive substances. Only the surface structure of the passive material can resist microbial contamination. Examples include “the lotus effect” and the micro-domain structured surface. The key is to make it impossible for microbial cells to adhere to the fibre’s surfaces. Active antimicrobial clothing contains bioactive substances which kill microbes (**Russell and Chopra 1996; Beumer et al. 2000).**

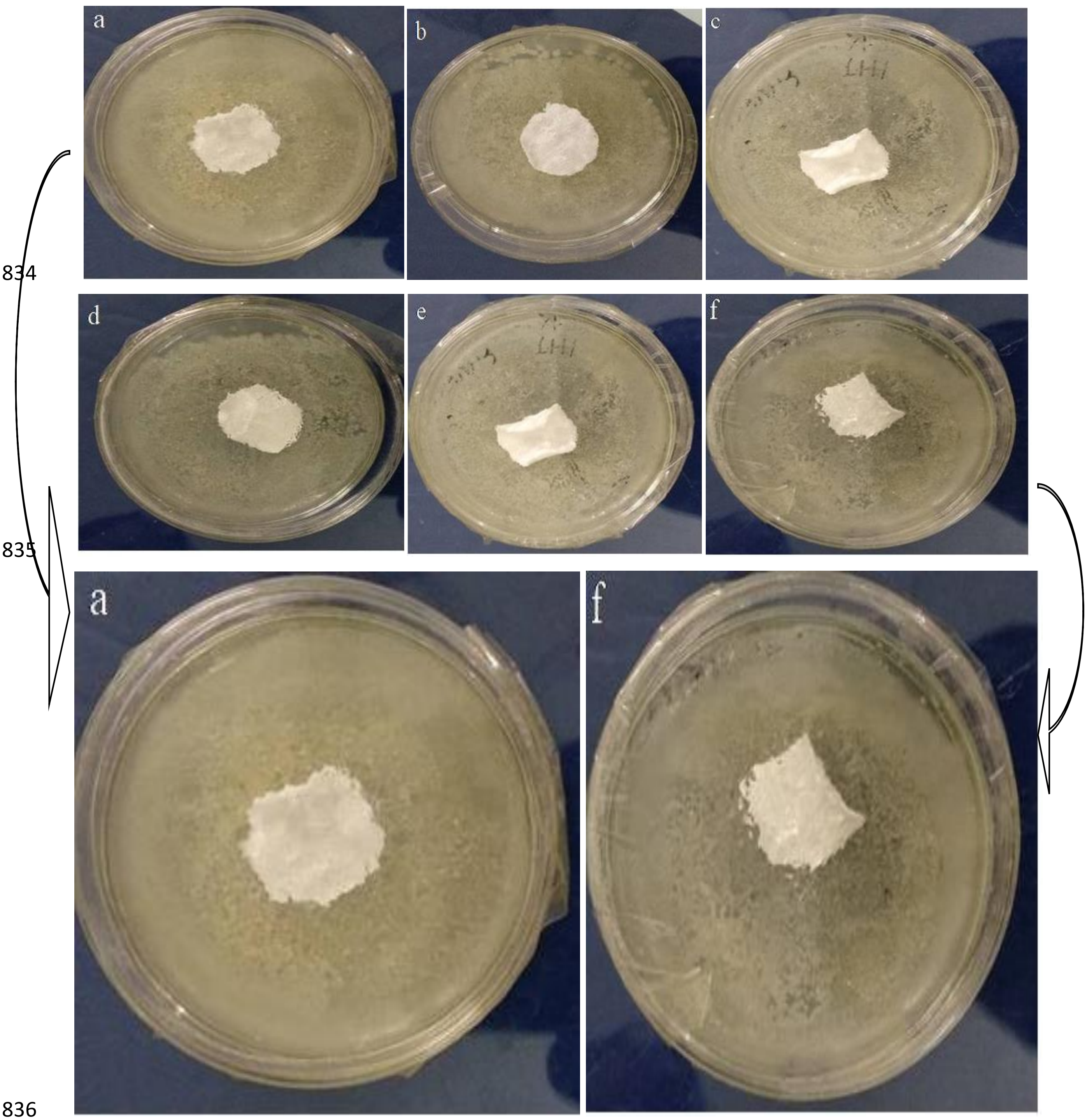


Fig. 15. Antibacterial activity of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric

Recently, dendrimers have been proposed to develop antimicrobial properties for applications to textiles. **Ghosh et al., (2010)** observed effective antimicrobial activities of the modified-dendrimer treated-fabric against *Staphylococcus aureus* (*S. aureus*).

Clear microbial inhibition zones were not found for untreated or treated fabrics in **Fig. 15 (a-f).** However, bacterial population growth seems to be less on the FC-dendrimer-treated sample in **Fig. 11(b-f)** than the untreated sample in **Fig. 11(a).** Among the treated samples, 100 g/L FC-dendrimer treated fabric **(Fig. 11(f))** showed the lowest bacterial population growth.

FC-dendrimer polymer has no active antimicrobial properties. But it can act as a passive antimicrobial agent, due to its conversion of fabric surfaces to super–hydrophobic. Hydrophobic and super-hydrophobic fabrics resist bacterial adherence to the fabric by making it a slippery surface like that of lotus leaves. Besides in this study, RUCO STAR EEE 6 was used, which contains additional dendrimers along with FC resin. It is assumed that the amino group (NH_2) in dendrimer is the responsible functional group for its antibacterial activity. After FC-dendrimer treatment, 100 g/L FC-dendrimer-treated samples exhibited maximum amount nitrogen (1.63%), as found by EDS analysis. The reason for a less antibacterial activity for treated samples, as the lesser amount of dendrimers present in FC resin.

GSM and thickness measurement

Table 9 Thickness and GSM of treated and untreated fabric.

Observation	Thickness (mm)	Thickness Increase (%)	GSM (g/m^2)	GSM Increase (%)
Untreated	0.48	-	180	-
60 g/L FC-dendrimer treated fabric	0.502	4.58	185	2.70
70 g/L FC-dendrimer treated fabric	0.506	5.83	187	3.74
80 g/L FC-dendrimer treated fabric	0.508	7.50	188	4.25
90 g/L FC-dendrimer treated fabric	0.522	8.75	190	5.26
100 g/L FC-dendrimer treated fabric	0.524	9.16	191	5.75

After FC-dendrimer treatment, GSM and thickness of the treated fabrics increased markedly compared to that of the untreated fabric. The FC-dendrimer resin closed all the pores in the fabric, creating a chemical coating. That was the reason behind the thickness and weight increases of the fabric. Both GSM and thickness gradually increased with increased FC-dendrimer resin concentration.

Bursting strength measurement

Table 10 Bursting strength test result

Observation	Bursting strength (Kpa)
Untreated fabric	176
60 g/l FC treated fabric	180
70 g/l FC treated fabric	183
80 g/l FC treated fabric	186
90 g/l FC treated fabric	189
100 g/l FC treated fabric	188

Bursting strength is the amount of pressure that ruptures the fabric. The bursting strength of single jersey knitted fabric was tested according to the ASTM (D 3786-87) method. The increase or decrease in bursting strength is mainly a function of the smoothness or roughness of the treated fabric's surface. Generally, Smooth surface increases bursting strength and vice versa. The SEM image revealed that micro-roughness was developed on fluorocarbon-treated cotton (**Fig. 15(b-f)**) in comparison to the untreated cotton fabric. Micro surface roughness increases with increases in the water contact angle of the treated fabric. But, visually FC-dendrimer treated cotton fabric showed a smooth surface. It is assumed that softening agents, like non-ionic polysiloxane, were the main contributor factor for visual smoothness and smooth hand feel of the treated fabric. In the present study **Table 10** shows that the bursting strength of treated fabric increases as the FC-dendrimer concentration increases. Finally, it can be said that the SEM result did not support the present outcome. The true explanation for this is still unclear.

Abrasion resistance measurement

The aim of this study was to see if the treated fabrics could withstand abrasion. The research was carried out on a cloth that was rubbed against itself. This was conducted on a Martindale machine where the expected results were that the treatment should be rubbed off to some extent. The spray rating remained unchanged after 5,000 rub cycles, according to the results (**Table 11**). On the other hand, water repellency was decreased a little bit after 10,000 rub cycle. Water spray rating of 60 g/L FC-dendrimer treated fabric was retained 4 according to ASTM D4966-98 (1989) method. After a 10,000 rub cycle, 70 g/L FC-dendrimer treated fabric maintains a 4-5 spray rating. 80, 90, 100 g/L FC-dendrimer treated fabrics showed the best abrasion resistance.

Table 11 Spray rating result after abrasion

Observation	Martindale rub cycle	Spray rating after abrasion	Martindale rub cycle	Spray rating after abrasion
60 g/l FC-D treated fabric	5000	5	10, 000	4
70 g/l FC-D treated fabric	5000	5	10,000	4-5
80 g/l FC-D treated fabric	5000	5	10,000	5
90 g/l FC-D treated fabric	5000	5	10,000	5
100 g/l FC-D treated fabric	5000	5	10,000	5

Thermal conductivity

Thermal conductivity is heat flow through a material. **Table 12** illustrates that the least conductivity existed in 100 g/L FC-dendrimer-treated fabric and maximum conductivity was observed in 60 g/L concentration. The thermal conductivity value gradually decreased as the thickness and GSM of the treated sample gradually increased. As FC-dendrimer concentration increased, the rate of heat transfer decreased. The thermal conductivity rate of 90 g/L and 100 g/L FC-dendrimer-treated fabric were decreased to 40% and 54% respectively, due to their greater thickness. Moreover, it is assumed that changing the surface morphology of the treated fabric also created barriers to thermal conductivity. SEM study revealed that the FC-dendrimer polymer not only filled the pores of the untreated fabrics but also exhibit small granular material on the surface of the treated fabric, which would reduce thermal conductivity in the treated sample.

Table 12 Thermal conductivity test results and their Chi-Square (χ^2) hypothesis test observation (Degree of freedom: (3-1) =2; Level of significance; 5%).

Observation	Thermal conductivity (Cal/cm/s/°C) $\times 10^{-3}$	Calculated value	Tabulated value	Comments
60 g/L FC-dendrimer treated fabric	17.8	2.73	5.99	Null hypothesis test is accepted
70 g/L FC-dendrimer treated fabric	16.53	4.46		Null hypothesis test is accepted
80 g/L FC-dendrimer treated fabric	15.63	5.98		Null hypothesis test is accepted
90 g/L FC-dendrimer treated fabric	13.43	10.81		Null hypothesis test is rejected
100 g/L FC-dendrimer treated fabric	10.26	19.46		Null hypothesis test is rejected

*Thermal conductivity of untreated fabric: 22.3 (Cal/cm/s/°c) $\times 10^{-3}$

The calculated values of 60 g/L, 70 g/L and 80 g/L FC-dendrimer-treated fabric are less than the tabulated values as shown in **Table 12**, so the null hypothesis is accepted. On the other hand, the calculated values of 90 g/L and 100 g/L FC-dendrimer-treated fabrics are greater than the tabulated values: as a result, the null hypothesis is rejected. Therefore, thermal conductivity will not be hampered by 60 g/L, 70 g/L and 80 g/L FC-dendrimer treatment. But the comfort properties of the fabric will be significantly and negatively affected by 90 g/L and 100 g/L FC-dendrimer treatment.

Air permeability

Comfort and breathability are closely related to air permeability. The air permeability rate of 60 g/L, 70 g/L, 80 g/L, 90 g/L and 100 g/L FC-dendrimer treated fabric was 829, 818, 790, 750 and 733 $m^2/m^3/h$, respectively, as shown in **Table 13.** The highest air permeability value existed in 60 g/L and the air permeability of 90 g/L and 100 g/L treated fabrics were 13% and 15% lower respectively, compared to that of untreated fabric. Among the FC-dendrimer-treated samples. As the concentration of FC-dendrimer coating materials increased, the pickup percentage gradually increased and, as a direct result, air permeability value declined. Cross-linking networks may form on the water-repellent finish. The FC-dendrimer treatment results in the blocking of some pores, which may be responsible for air permeability being less. These are some of the factors that may create less air permeability.

Table 13 Air permeability test results and their Chi-Square (χ^2) hypothesis test observation (Degree of freedom: 3-1 = 2; and Level of significance; 5%).

Observation	Mean value of Air Permeability rate in (m^3/m^2/hour)	Chi-square calculated value	Chi-square Tabulated value	Comments
60 g/L FC-dendrimer treated fabric	829	3.29		Null hypothesis test is accepted
70 g/L FC-dendrimer treated fabric	818	5.97		Null hypothesis test is accepted
80 g/L FC-dendrimer treated fabric	790	16.78	5.99	Null hypothesis test is rejected
90 g/L FC-dendrimer treated fabric	749	42.46		Null hypothesis test is rejected
100 g/L FC-dendrimer treated fabric	732	56.56		Null hypothesis test is rejected

Air permeability of untreated fabric: 860 (m^3/m^2/hour)

The critical chi-square value is χ^2 at a 5% level of significance and the degree of freedom is 2 as three observations were taken for each sample. The calculated values for 60 g/L and 70 g/L FC-dendrimer-treated fabrics are less than the tabulated values then the null hypothesis is accepted as shown in **Table 7.** On the other hand, the calculated value for 80 g/L, 90 g/L and 100 g/L is greater than the tabulated value, the null hypothesis is rejected as shown in **Table 7.** The 80 g/L, 90 g/L and 100 g/L FC-dendrimer-treated fabric observation did not achieve a sufficient value for acceptance of the null hypothesis but the 60 g/L and 70 g/L FC-dendrimer-treated fabric observation did. Thus, we conclude that 60 g/L and 70 g/L FC-dendrimer treatment will not hamper air permeability or comfort of the fabric. But this will not hold true at higher levels of FC-dendrimer concentration.

Water vapor permeability

Clothing should be able to let water vapor pass through it. Otherwise, heat accumulates in the body as humid water vapor is impacted between clothing and the body. Similarly, perspiration should pass through clothing. Lighter fabrics (less mass per square meter and thickness) permit the easy passage of water vapor through the fabrics.

The rate of water vapor permeability decreased with increased concentration of FC-dendrimer, as shown in **Table 14.** The water vapor permeability of 60 g/L, 70 g/L, 80 g/L, 90 g/L, and 100 g/L FC-dendrimer-treated fabrics were 1249, 1240, 1231, 1221 and 1212 gm/m^2/day, respectively.

Table 14 The results of water vapor permeability readings with variation of C6-FC-dendrimers.

Observation	After 01 hour in gm/m^2	After 02 hour in gm/m^2	After 03 hour in gm/m^2	After 04 hour in gm/m^2	After 24 hour gm/m^2
60 g/L FC-dendrimer treated fabric	41	85	142	222	1249
70 g/L FC-dendrimer treated fabric	38	83	139	219	1240
80 g/L FC-dendrimer treated fabric	37	80	137	217	1231
90 g/L FC-dendrimer treated fabric	35	78	135	213	1221
100 g/L FC-dendrimer treated fabric	32	75	133	207	1212

*Water vapor permeability of untreated fabric - 1288 gm/ m^2/day

Table 15 Chi-Square (χ^2) hypothesis test observation in terms of water vapor permeability value of treated fabric (Degree of freedom: (3-1) = 2; Level of significance, 5%).

Observation	Calculated value	Tabulated value	Comments
60 g/L FC-dendrimer treated fabric	3.6	**5.99**	Null hypothesis test is accepted
70 g/L FC-dendrimer treated fabric	5.51		Null hypothesis test is accepted
80 g/L FC-dendrimer treated fabric	7.65		Null hypothesis test is rejected
90 g/L FC-dendrimer treated fabric	10.56		Null hypothesis test is rejected
100 g/L FC-dendrimer treated fabric	13.69		Null hypothesis test is rejected

Water vapor permeability of untreated fabric: 1288 gm/ m^2/day

The calculated values of 60 g/L and 70 g/L FC-dendrimer-treated fabrics are less than the tabulated value, so the null hypothesis is accepted as shown in **Table 15**. On the other hand, the calculated values of 80 g/L, 90 g/L and 100 g/L FC-dendrimer-treated fabrics are greater than the tabulated values, as a result, the null hypothesis is rejected. It may be concluded that 60 g/L and 70 g/L FC-dendrimer treatment will not hamper the water vapor permeability rate as well as comfort properties. But comfort properties are greatly impaired by 80, 90 and 100 g/L FC-dendrimer treatment.

Conclusion

The purpose of this experiment was to test whether FC-dendrimer treatment could improve the characteristics of cotton fabric to make more functional as well as protective fabrics. In that sense, this experiment was a success. FC-dendrimer and crosslinking agents produce a fabric that is water, oil and dirt repellent, as well as UV-protective, flame retardant and antimicrobial. As the amount of FC-dendrimer concentration increases, the repellence of the treated fabric increases. An increase in the surface hydrophobicity, reduction of surface energy and lowering of the number of hydroxyl functional groups on the surface of cotton fabric are achieved by FC-dendrimer coating, enhancing its repellence and allowing unwanted water, oil and dirt to slide off, so that the fabric self-cleans. High contact angle values for liquids mean that FC-dendrimer-treated cotton fabrics have become hydrophobic. Achieving such a hydrophobic character, the treated fabric efficiently separated water/oil mixtures on the fabric. Results showed that fabric coated with 60, 70, 80 g/L FC-dendrimer displayed the best performance in comfort. On the other hand, 90 and 100 g/L showed the best result in terms of water resistance. Therefore, FC-dendrimer treatment can produce textiles which are more suitable than untreated cotton for a wide range of functions, including anti-wetting, self-cleaning, oil-water separation, antimicrobial articles and protective textiles.

Limitations

In this study, spray rating did not change after five times machine wash. On the other hand, the spray rating did not change after the 5000 rub cycle. Furthermore, 70, 80 and 100 g/L FC-dendrimer treated fabric still maintained their hydrophobicity even after the 10,000 rub cycle. Therefore, it can be said that self-cleaning, oil-water separation, hydrophobic, antibacterial, UV resistant and flame retardant properties still retained their performance after five-time machine wash and 10,000 rub cycle. All the self-cleaning, oil-water separation, antibacterial, UV resistant and flame retardant properties mainly depend on the hydrophobic performance of the fabric for this study. Some literature revealed that fluorocarbon treated fabric has become unusable after 50 (fifty) washing cycles and 50,000 rub cycles. So it needs to be further investigated. Several types of dendrimer are available in the market. In this study FC-dendrimer (Rucostar EEE6, Rudlof, Germany) has been used. The detailed mechanism of FC-dendrimer did not found. For this reason, cross-linking network between FC-dendrimer and the cotton fabric did not possible to demonstrate. Furthermore, the mechanism of UV resistivity did not explain properly due to a lack of detailed information on the FC-dendrimer chemical structure. These will be of great interest to researchers for future investigation.

Compliance with ethical standards

Conflicts of interest

The authors declare they have no conflicts of interest.

Ethical standard

This articles does not involve the participation of humans or animals.

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Figures

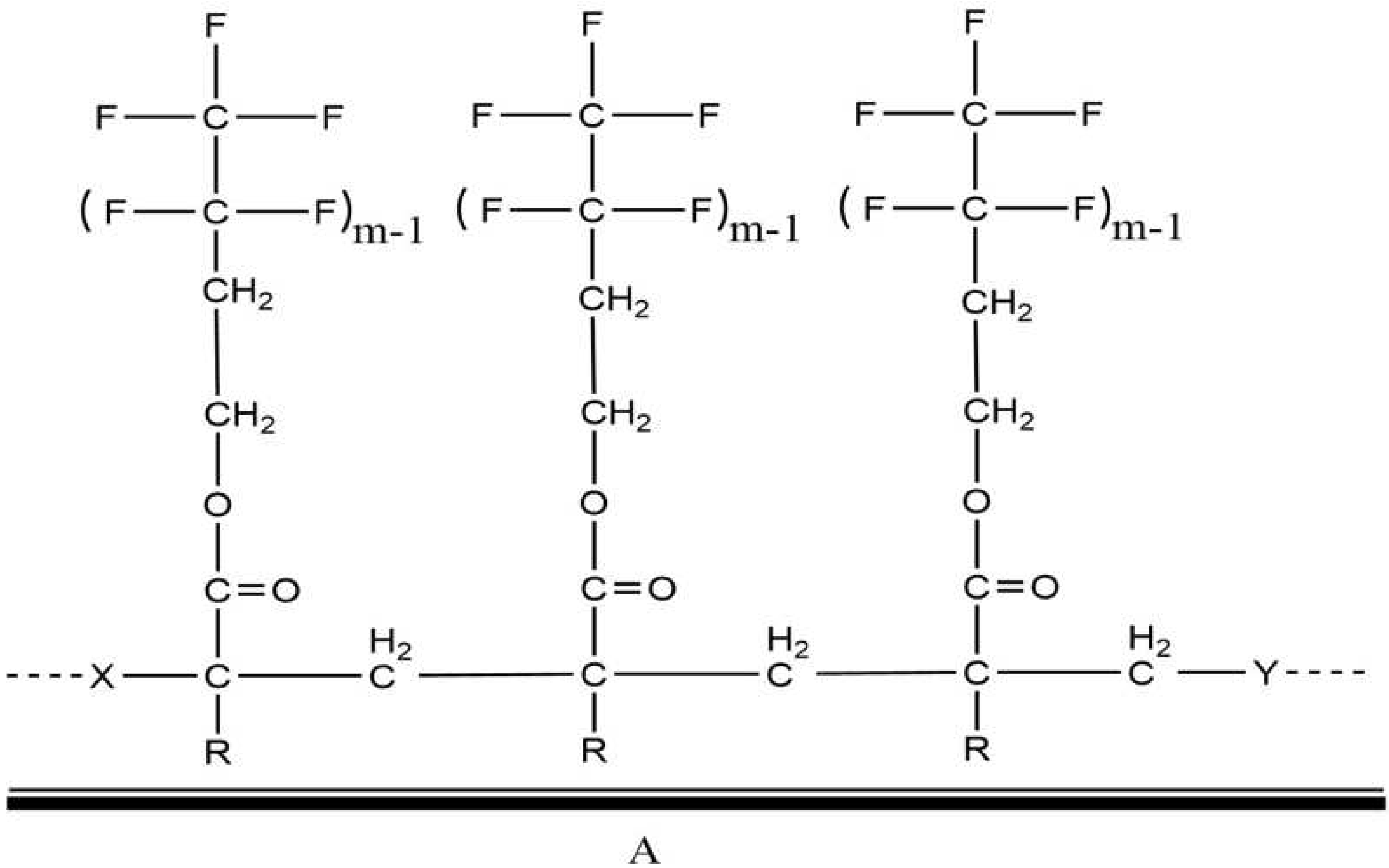

Figure 1

Fluorocarbon polymer on fibre surface. Where m=6-10, X and Y are co-polymers mainly stearylacrylates, R= H or CH3 (polyacrylic or polymethacrylic acid esters), and A is the fire surface.

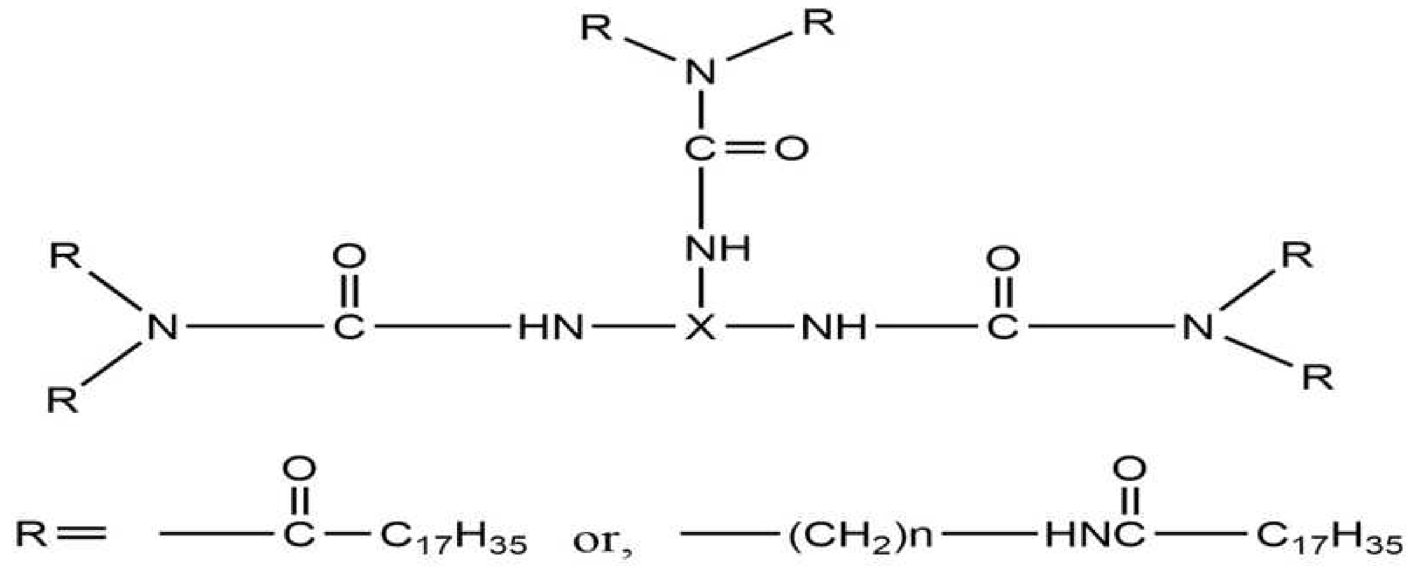

Figure 2

A dendrimer structure synthesized from three distearyl-amines or amides and a tri-functional isocyanate X (N=C=O).

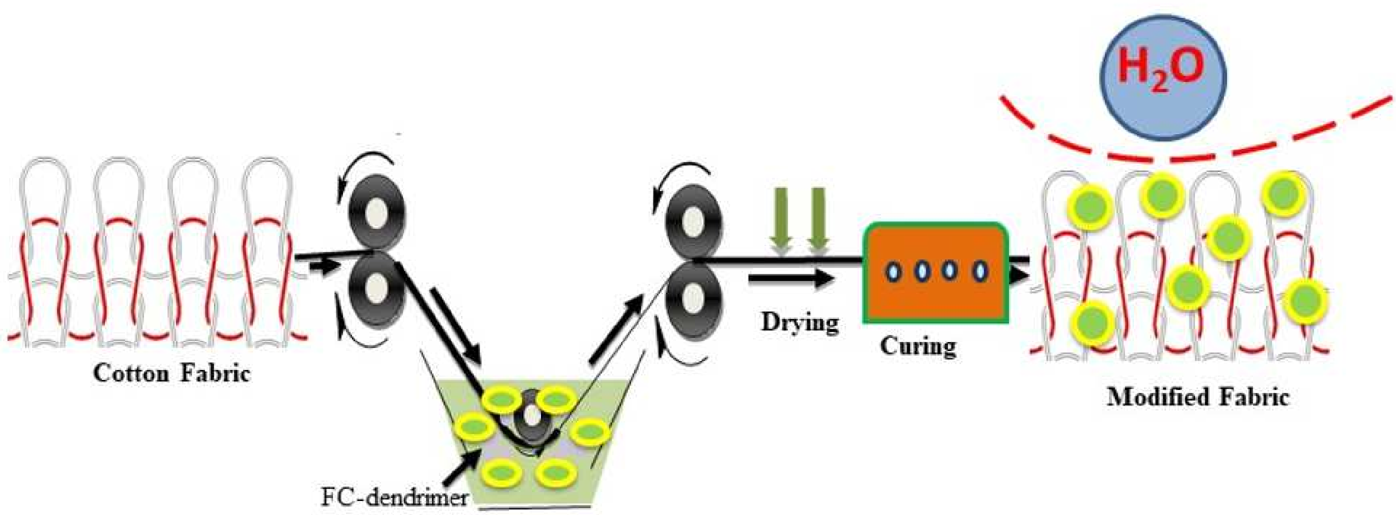

Figure 3

Schematic diagram of Sample preparation process

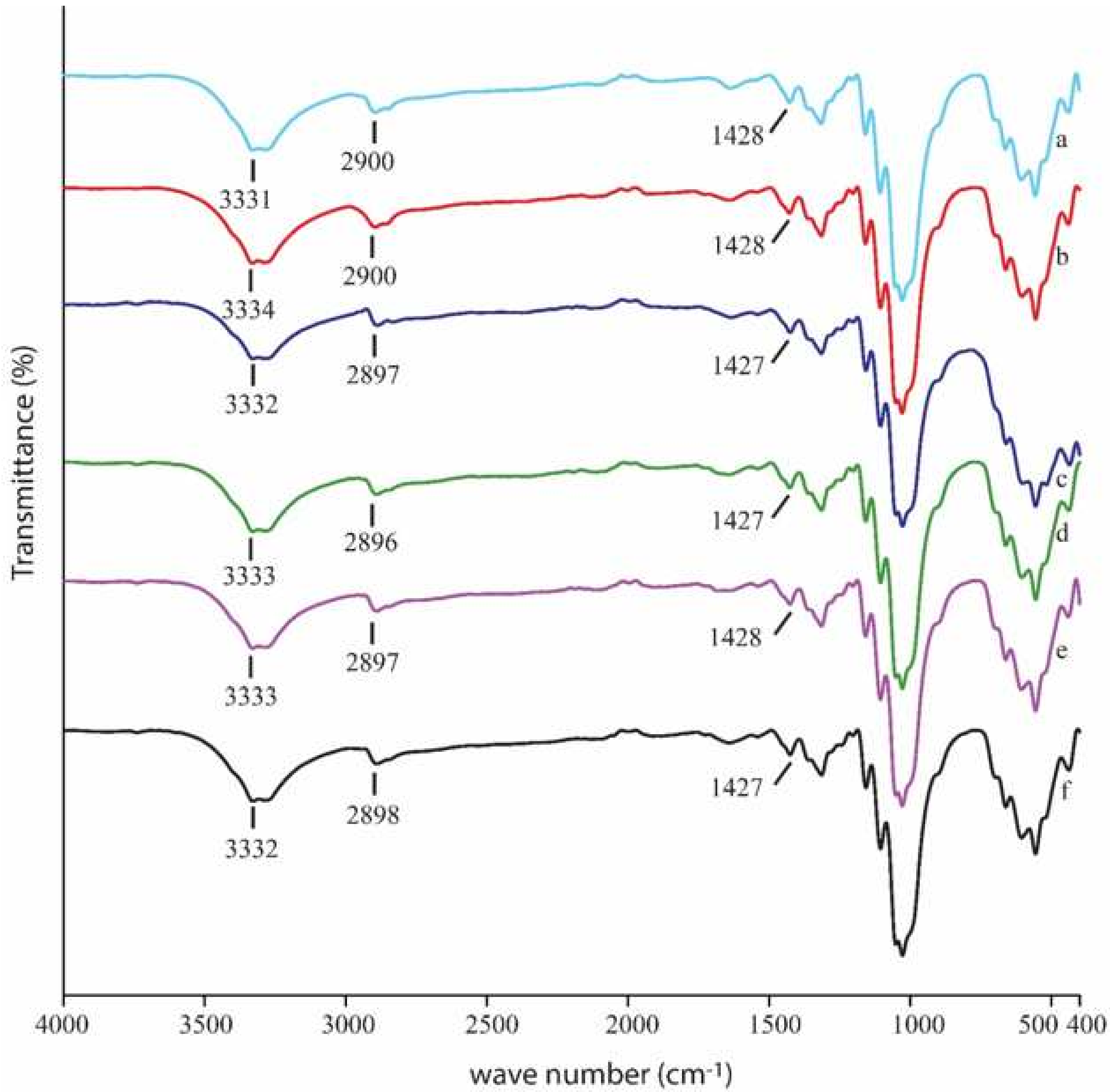

Figure 4

FTIR spectra of (a) Untreated fabric, (b) 60 g/L FC-dendrimer, (c) 70 g/L FC-dendrimer, (d) 80 g/L FC-dendrimer, (e) 90 g/L FC-dendrimer and (f) 100 g/L FC-dendrimer treated fabric.

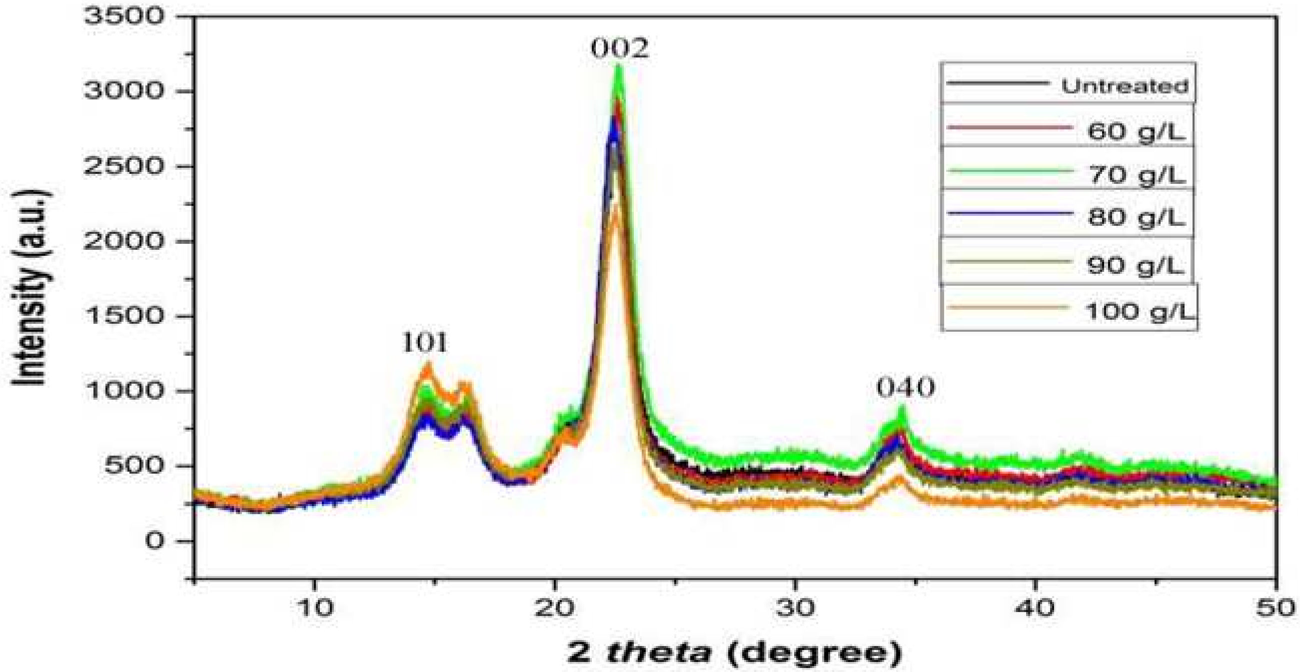

Figure 5

X-Ray Diffraction pattern untreated and FC-dendrimer treated fabrics.

Scanning electron microscopy (SEM) study

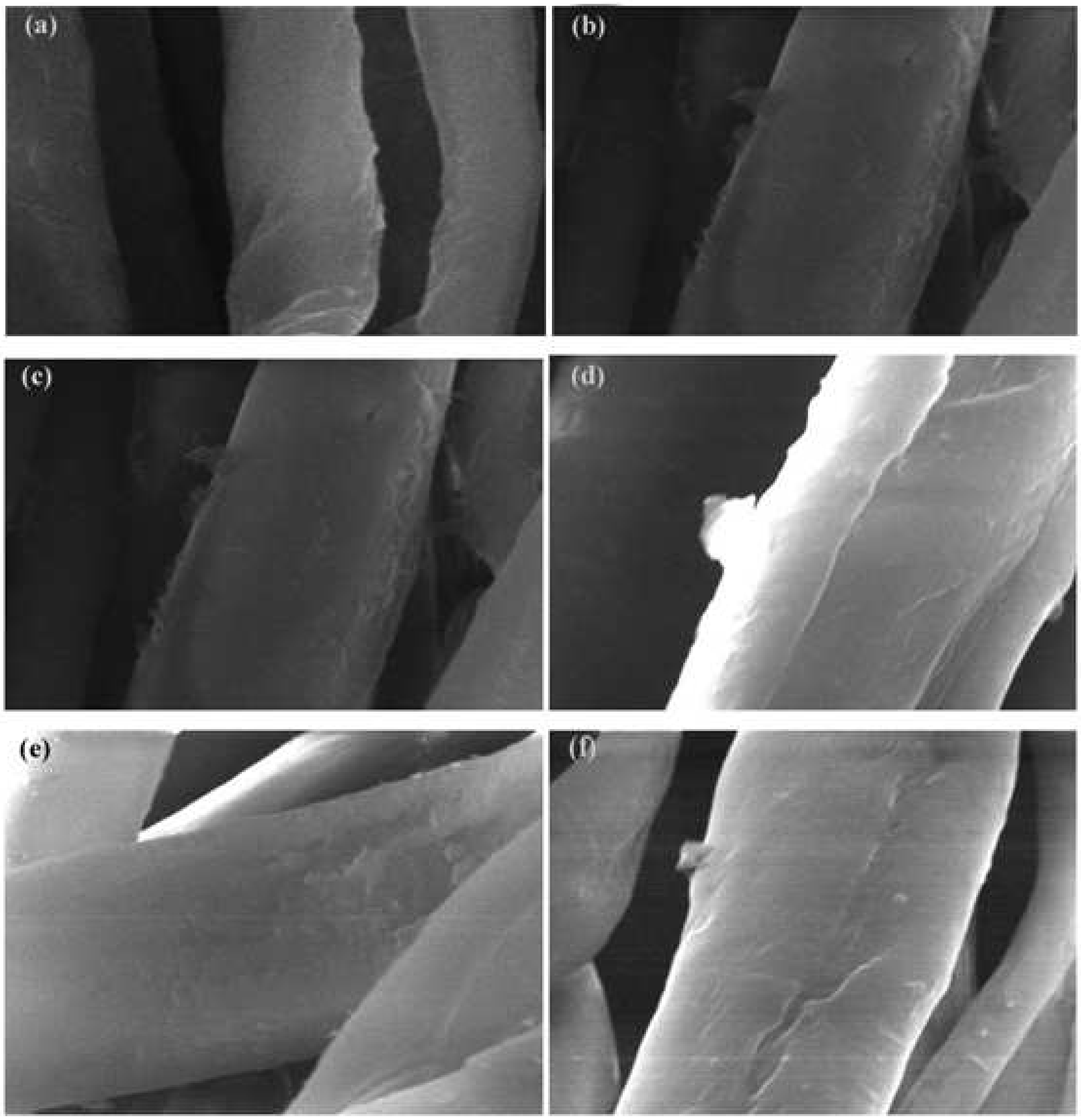

Figure 6

SEM photograph at × 3000 magnification and 1μm scale of (a) Untreated fabric, (b) 60 g/L FC-dendrimer, (c) 70 g/L FC-dendrimer, (d) 80 g/L FC-dendrimer, (e) 90 g/L FC-dendrimer and (f) 100 g/L FC-dendrimer treated fabric.

EDS Analysis

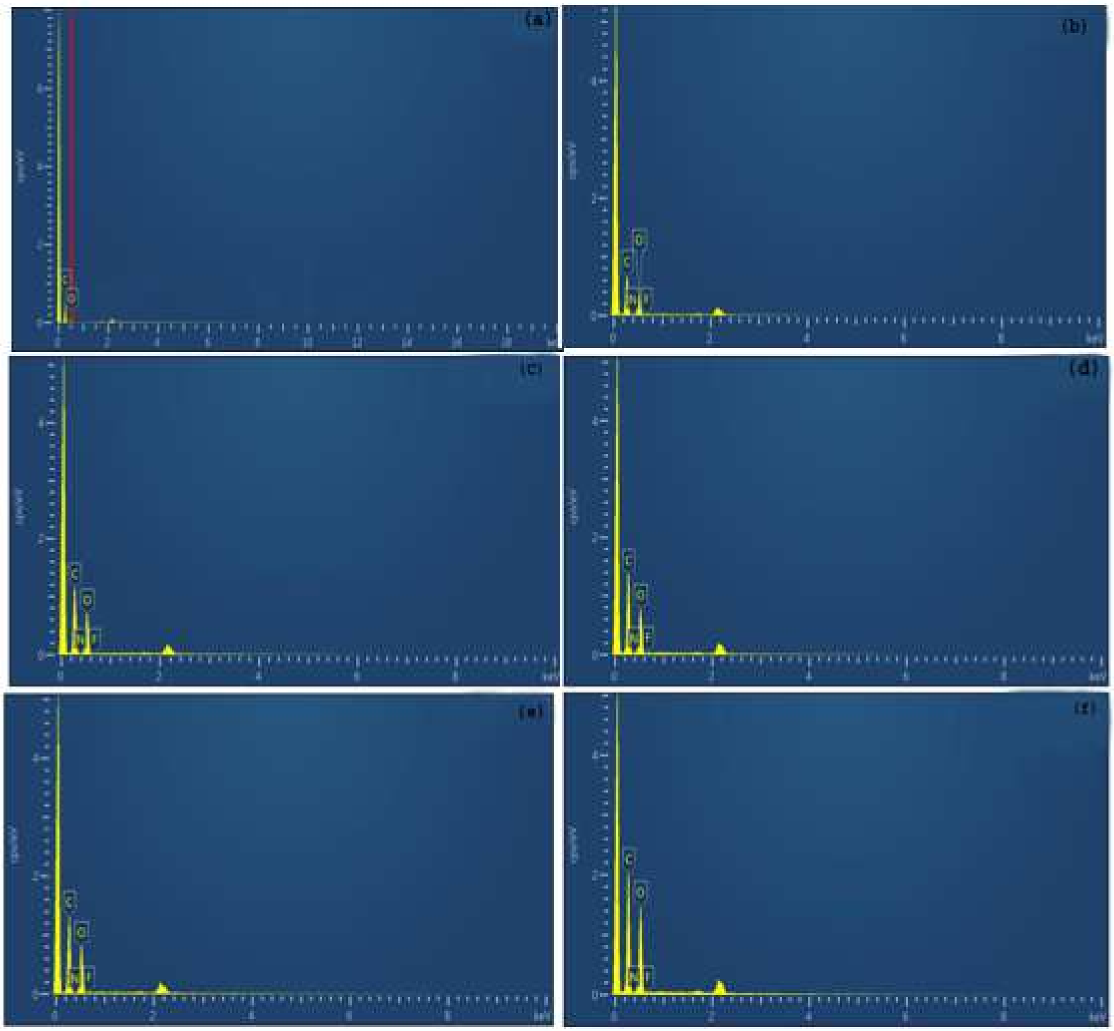

Figure 7

EDS photograph of (a) Untreated fabric, b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

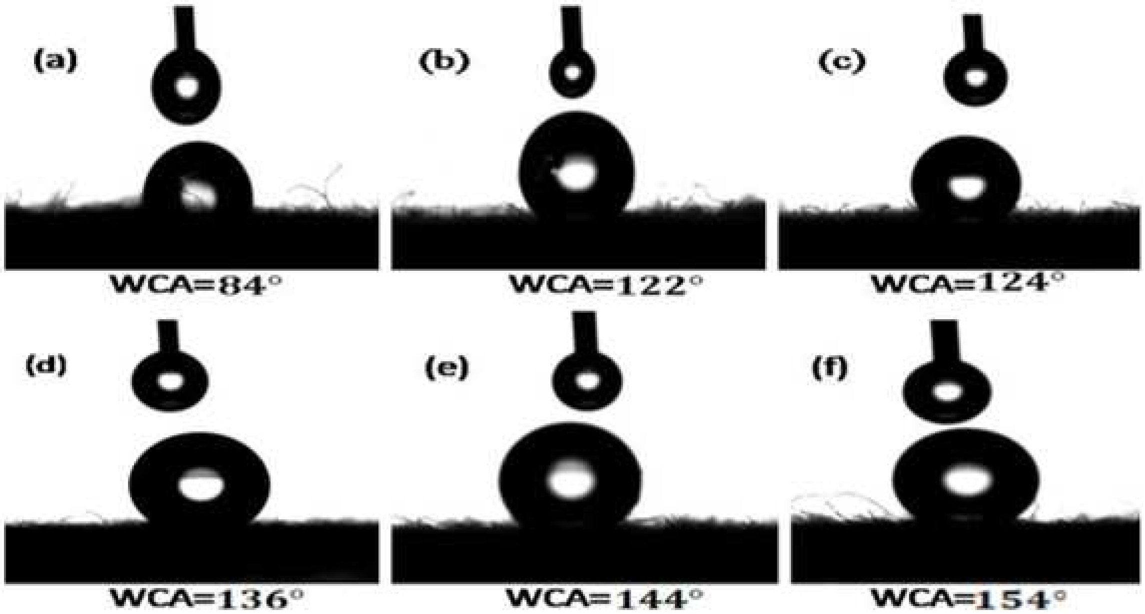

Figure 8

Water contact angle (WCA) of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

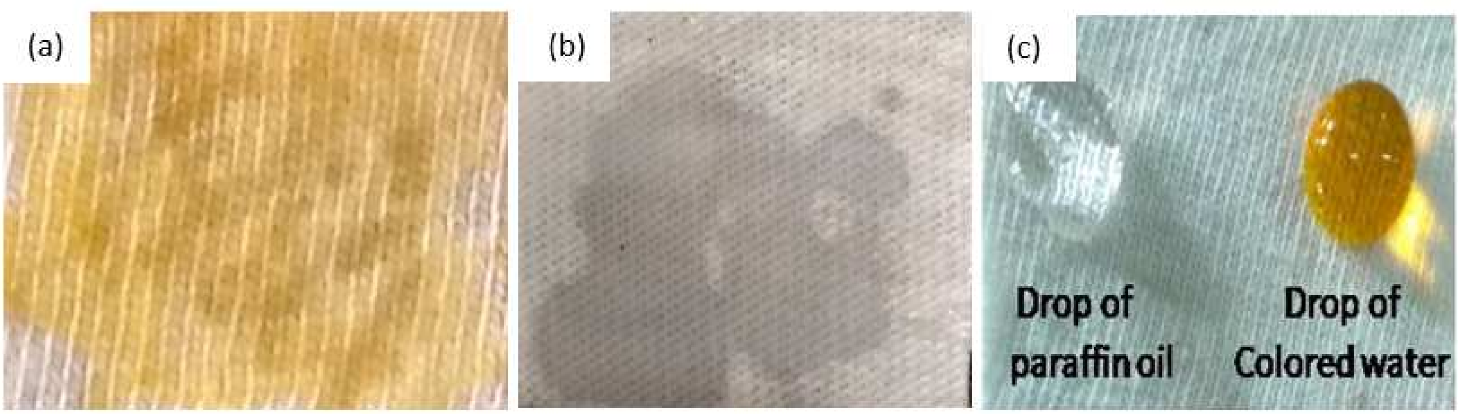

Figure 9

Water-oil repellent properties of (a) & (b) Untreated fabric and (c) 100 g/L FC-dendrimer treated fabric.

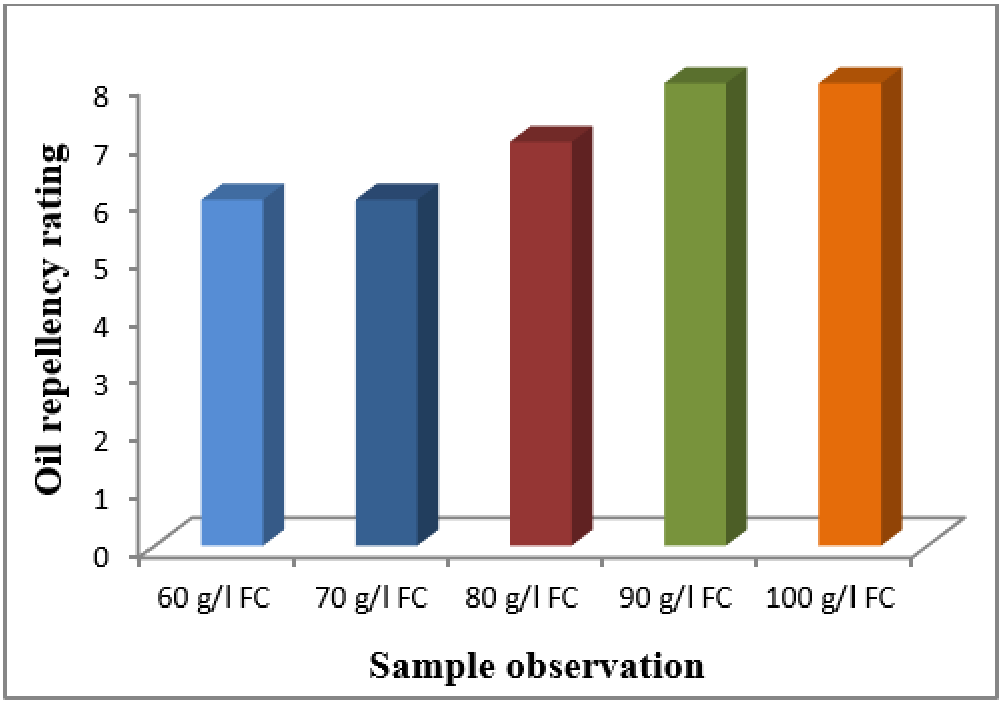

Figure 10

Oil repellency rating of the FC-dendrimer treated sample.

Figure 11

Schematic diagram of oil-water separation process.

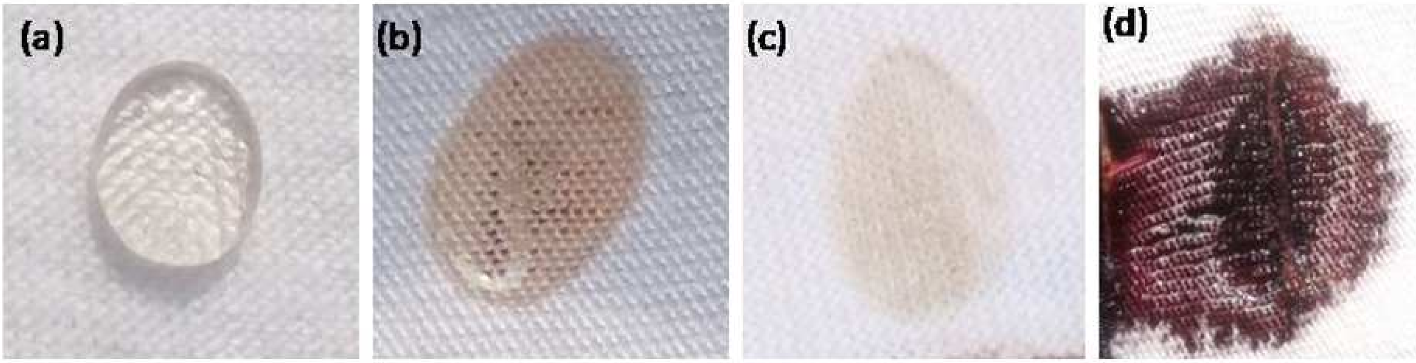

Figure 12

Acid resistance performances of (a) 100 g/L FC-dendrimer treated fabric after one 1 min., (b) Untreated fabric after one 1 min., (c) 100 g/L FC-dendrimer treated fabric after 30 min. and (d) untreated fabric after 30 min.

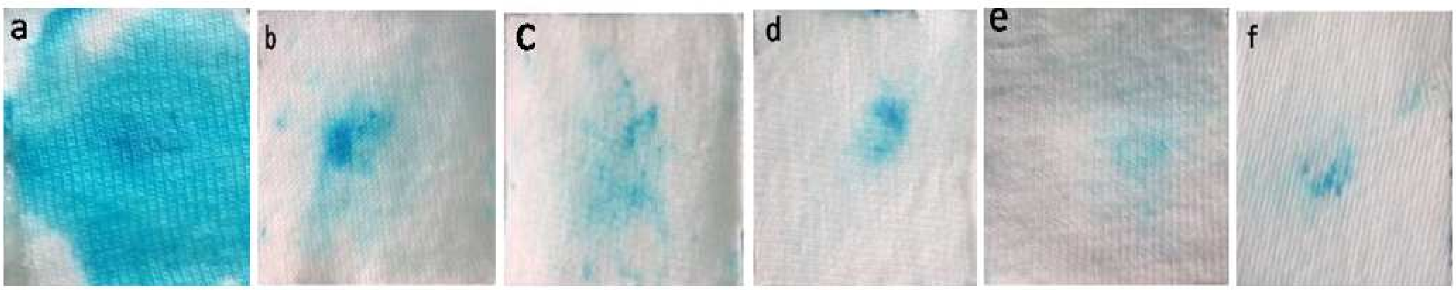

Figure 13

Self-cleaning performances of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric.

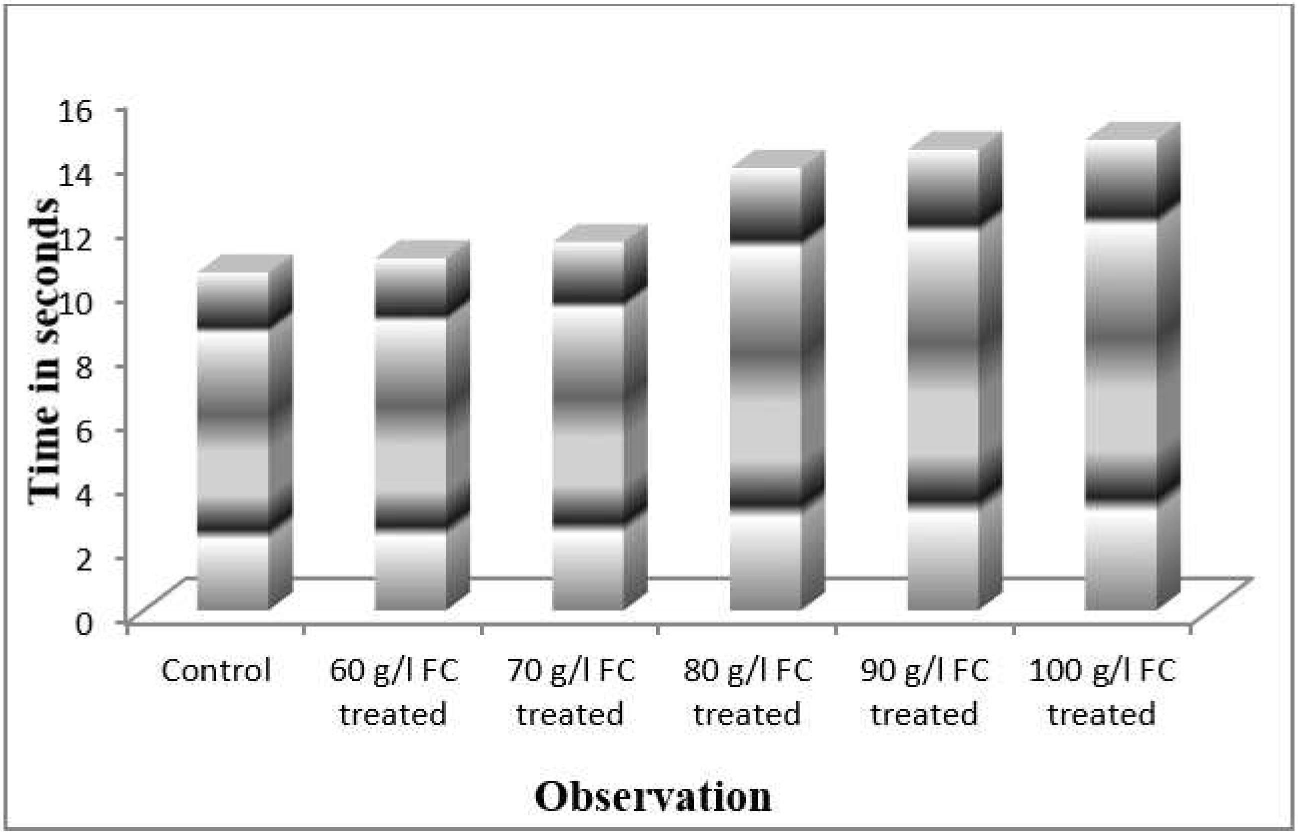

Figure 14

Burning required time of untreated and treated fabric.

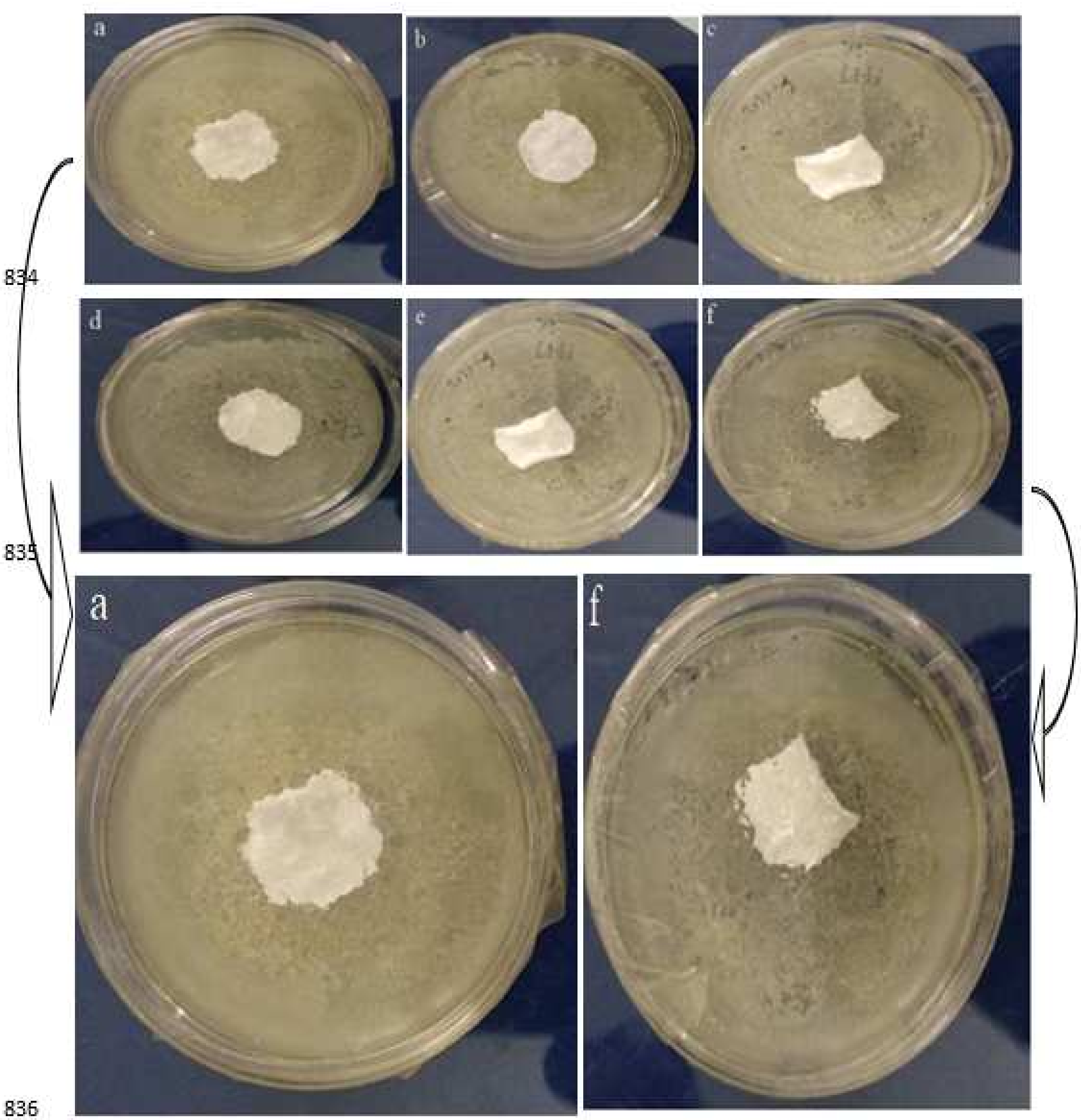

Figure 15

Antibacterial activity of (a) Untreated fabric, (b) 60 g/L FC-dendrimer treated fabric, (c) 70 g/L FC-dendrimer treated fabric, (d) 80 g/L FC-dendrimer treated fabric, (e) 90 g/L FC-dendrimer treated fabric and (f) 100 g/L FC-dendrimer treated fabric

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- floatimage1.jpeg